

Serial No
920299

**UV/Visible 9420/9430
Spectrophotometer
User's Manual**

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PREFACE

This manual provides the information needed to properly install and operate the IBM Instruments Models 9420 and 9430 Ultra-violet/Visible Spectrophotometers. The installation instructions are provided in Section 1.0. A general operating description is given in Section 2.0. Sections 3.0 - 14.0 contain a detailed description of the various operating modes of these instruments. Each procedure is described in a step-by-step style which allows a rapid familiarization with the features and capabilities of both the 9420 and the 9430.

Related Publications

9400 Series Product Description GC22-9216

9410 Spectrophotometer User's Manual, GC22-9218

9400 Series Supplies and Accessories Catalog, GC22-9290

9400 Series Service Manual GC22-9219 (To be supplied)

9400 Series Parts Catalog GC22-9220 (To be supplied)

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1.0 INSTALLATION

1.1 INTRODUCTION

The IBM Instruments 9420 and 9430 are double-beam, scanning, ultra-violet/visible spectrophotometers. These instruments are designed for the accurate measurement of either absorbance or transmittance in the 900-195nm range.

Both the 9420 and 9430 are bench-top instruments consisting of two main components: an optical unit and an operator's console as shown in Figure 1-1.

The instrument is controlled from the operator's console. The keypad is used to enter commands and change instrumental parameters. A high-resolution CRT display provides prompting during parameter entry, and data display. A built-in thermal printer/plotter is used to obtain a permanent record of spectral data and operating conditions.

The optical unit contains the monochromator, light sources, detector and other optical components needed to perform spectrophotometric measurements. The sample is mounted in the sample chamber in the center of the optical unit. A sliding door at the top of the sample chamber provides easy access to the cell holder. The entire sample chamber can be removed from the optical unit by loosening two thumbscrews under the front edge of the chamber, and by pulling the chamber up and forward. This allows a variety of accessories to be mounted in the optical unit.

The operator's console and optical unit are connected via a cable that attaches to the rear panel of each unit. In the sections that follow, a detailed installation procedure is described.

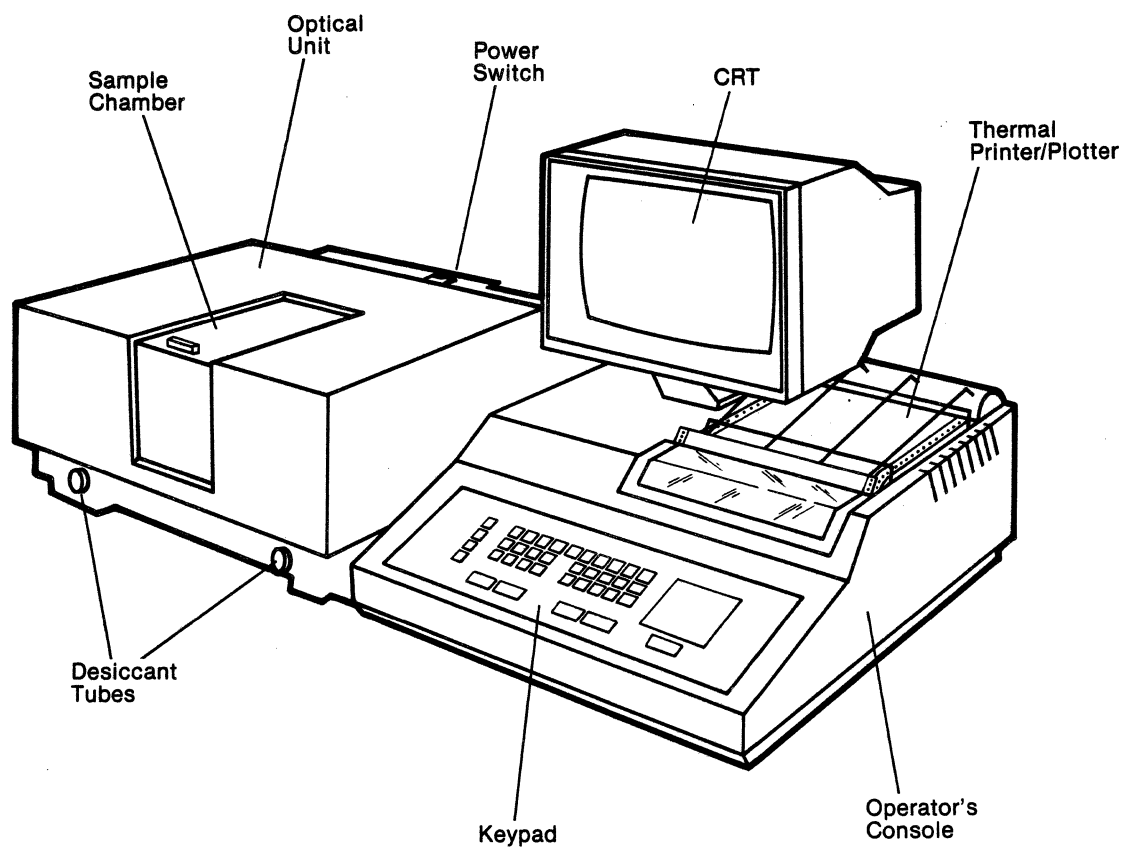


Figure 1-1. Model 9420/9430 Ultra-Violet/Visible Spectrophotometer

1.2 ENVIRONMENTAL REQUIREMENTS

The 9420 and 9430 spectrophotometers have been designed to operate under normal laboratory conditions. The following guidelines should be used to select an appropriate area for installation of the instrument.

1. Temperature (Ambient) : 15°C (59°F) to 35°C (95°F).
2. Relative Humidity : 35% to 85%.
3. Atmospheric Pressure : 760 to 1,060 millibars.
4. Select an area of operation that is not in direct sunlight or close to an instrument or machine that generates strong magnetic fields or vibrations. Avoid areas where dust and corrosive gases might be present.
5. Do not install the spectrophotometer near an air conditioner vent.
6. Provide a flat, firm surface (approximately 4 feet wide by 3 feet deep) that can support 150 pounds. Ideally, there should be enough space in back of the instruments to allow connections to be made easily, and for inspection and maintenance of the spectrophotometer components.
7. Optical Unit Dimensions :

	<u>9420</u>	<u>9430</u>
Width:	53 cm (21 in)	53 cm (21 in)
Depth:	58.5 cm (23 in)	61 cm (24 in)
Height:	25.5 cm (10 in)	25.5 cm (10 in)
8. Optical Unit Weight : 40 kg (88 lbs).
9. Operator's Console Dimensions :

Width:	55 cm (21.5 in)
Depth:	63.5 cm (25 in)
Height:	47 cm (18.5 in)
10. Operator's Console Weight : 23 kg (51 lbs).

1.3 ELECTRICAL REQUIREMENTS

Input Voltage : 120 Volts, AC (117.7 VRMS \pm 10%)
single phase, grounded supply.

Frequency : 60 Hz.

Power Consumption : 500 VA nominal.

NOTE: The following precautions should be observed in setting up and operating these instruments.

1. Source Power

Facilities wiring and wiring devices should be in compliance with the National Electrical Code, Underwriters Laboratory, and any local electrical installation codes that apply at your facilities. If any doubt exists regarding compliance, your local power company or electrical contractor can provide assistance.

A grounded receptacle that has a 115VAC output must be located within three feet of the spectrophotometer. A line that is fused for 20 amps will supply ample power for all instruments.

2. Instrument Enclosure

These Safety Class 1 instruments are designed to operate with all covers and panels in place. The covers serve two purposes beyond enclosing the instrument: they provide safety for personnel and they form a part of the noise immunity system.

3. Grounding AC Safety

The instruments have an AC grounding system, the primary purpose of which is to provide electrical safety. The AC grounding system is readily identified by the green-yellow tracer wires. These wires should never be removed, nor should the AC grounding configuration be altered. There should be very low impedance, less than 0.2 ohms, for every external conductive surface on the instrument, back to facilities ground and the ground pin on the plug.

1.4 UNPACKING AND INSPECTION

All instruments are carefully checked before leaving the factory. When the equipment is received by the user, it should be checked to ensure that it has not been damaged in transit.

The 9420/30 UV/VIS Spectrophotometers are shipped in three cartons containing the Optical Unit, the Operator Control Unit, as well as the CRT and a box of supplies.

1. Make sure all cartons are sitting right side up as indicated by the lettering and symbols on the sides of the cartons.
2. With a utility knife, carefully cut the tape that bonds the flaps of each carton. It is suggested that the cartons and the packing material in them be saved, in case the instruments need to be shipped at a later date.
3. Clear an area approximately 4 feet wide by 3 feet deep on a flat, firm surface. Make sure that the supporting frame can safely support 150 pounds. Ideally, there should be enough space in back of the instruments to allow connections to be made easily, and for inspection and maintenance of the spectrophotometer components.
4. Remove the packing material and, with the help of another person, lift the Optical Unit, the Operator Control Unit and the CRT from their boxes and place them in the prepared area.
5. To mount the CRT display, loosen the mounting screws located on top of the operator's console (approximately 9 turns of the screw). Slide the washers toward the head of each screw.

With the CRT tilted toward the back of the control unit, slide the slotted metal bracket over the mounting screws. (See figure 1-2.) Pull the CRT forward on the pedestal to engage the plastic retainer lip on the top of the console. The CRT should be sitting flat on the pedestal, so that the screen is nearly vertical (not tilted) when viewed from the side.

Secure the CRT by tightening the two mounting screws.

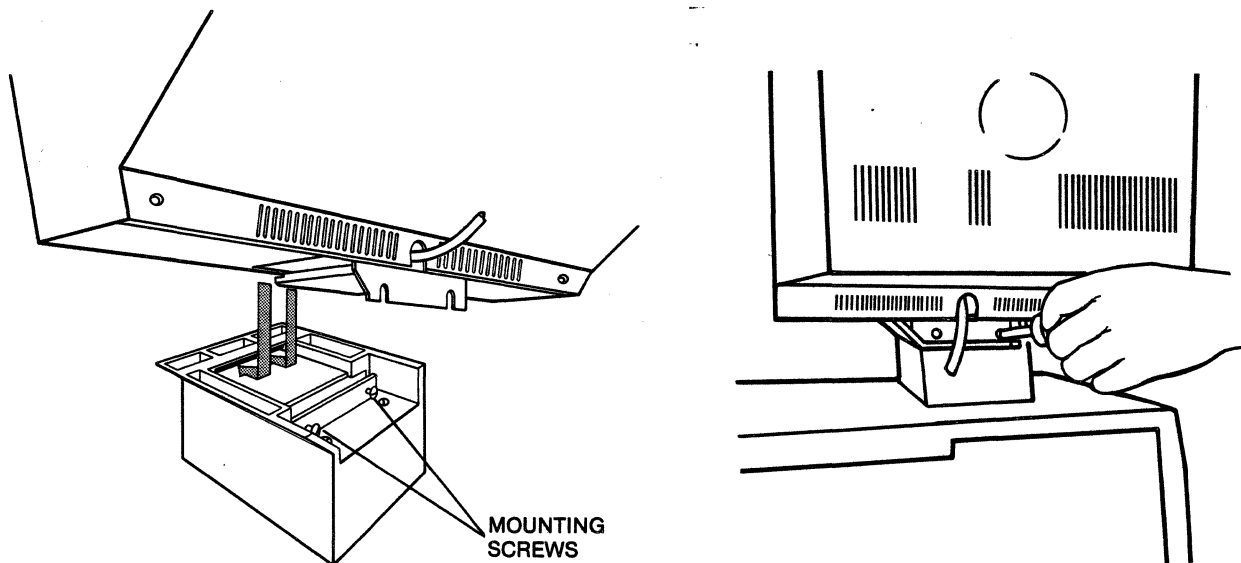


Figure 1-2. Mounting the CRT on the Operator's Console.

6. Press the rocker power switches at the rear of the Optical Unit and the Operator Control Unit to the OFF position.
7. Open the box placed in the CRT carton and verify that the following items are present:
 - ✓ 2 rectangular quartz cells, 10 mm path length
 - ✓ 1 holmium glass holder
 - ✓ 1 shutter plate
 - ✓ 1 AC power cord
 - ✓ 1 roll of thermal paper
 - ✓ 1 set of repair tools
 - 2 fuses
 - ✓ 1 bottle of silica gel
 - ✓ 1 dust cover
 - ✓ 1 control cable with identical multipin D - shaped connectors on each end
 - ✓ 1 paper guide
 - ✓ 2 thermal paper roll holder caps
 - ? 1 thermal paper separator and holder

1.5 SETUP

1. Locate the control cable with identical multi-pin "D" shaped connectors on each end. Use this cable to connect the Control jack on the rear panel of the optical unit to the Control jack on the rear panel of the operator's console.
2. Connect the cord attached to the CRT to the jack marked CRT at the rear of the Operator's Console.
3. Connect the black AC power cord that is attached to the rear of the Operator's Console to the AC 115V OUTLET at the rear of the Optical Unit.

Caution: The Operator's Console must be plugged into the Optical Unit for proper performance. Do not plug the Operator's Console directly into a wall outlet.

4. Connect the 16-pin connector attached to a black cord originating from the bottom of the optical unit to the JP 004 jack on the back of the same unit.
5. Connect the gray power cord to the 115 VAC IN on the back of the optical unit. Plug in the 3-prong connector to the wall outlet.
6. Figure 1-3 shows a rear view of the instrument after properly attaching all cables.

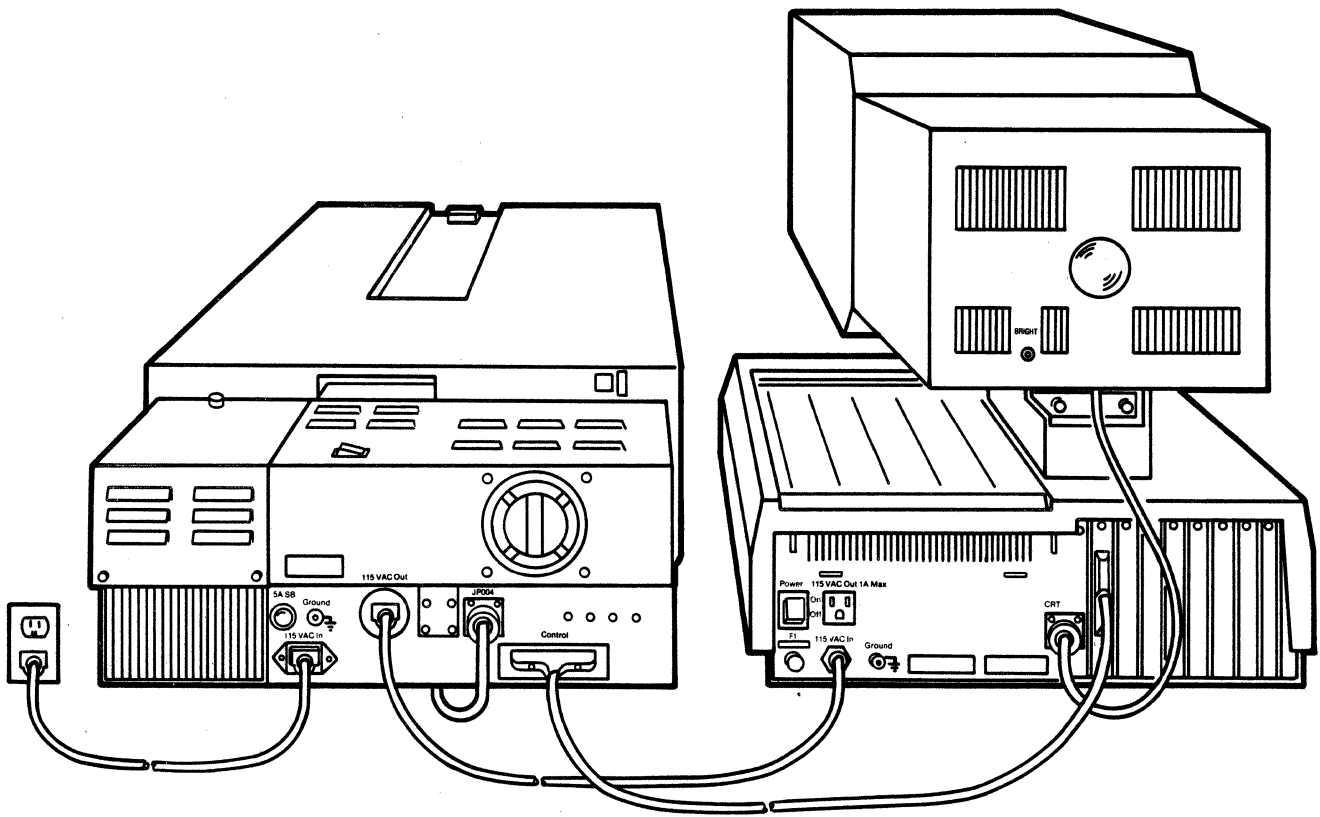
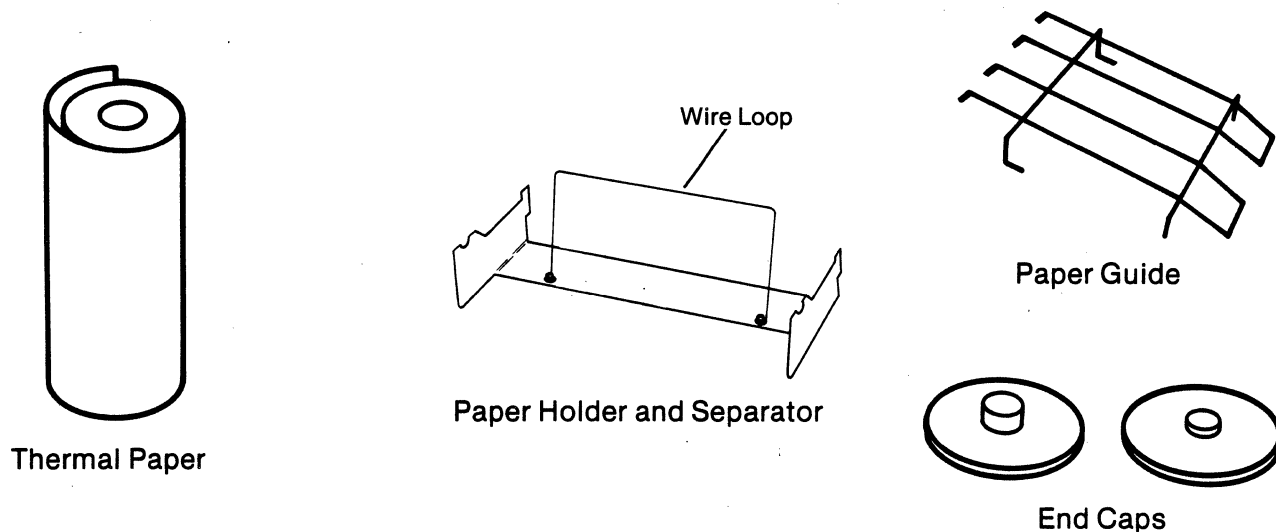


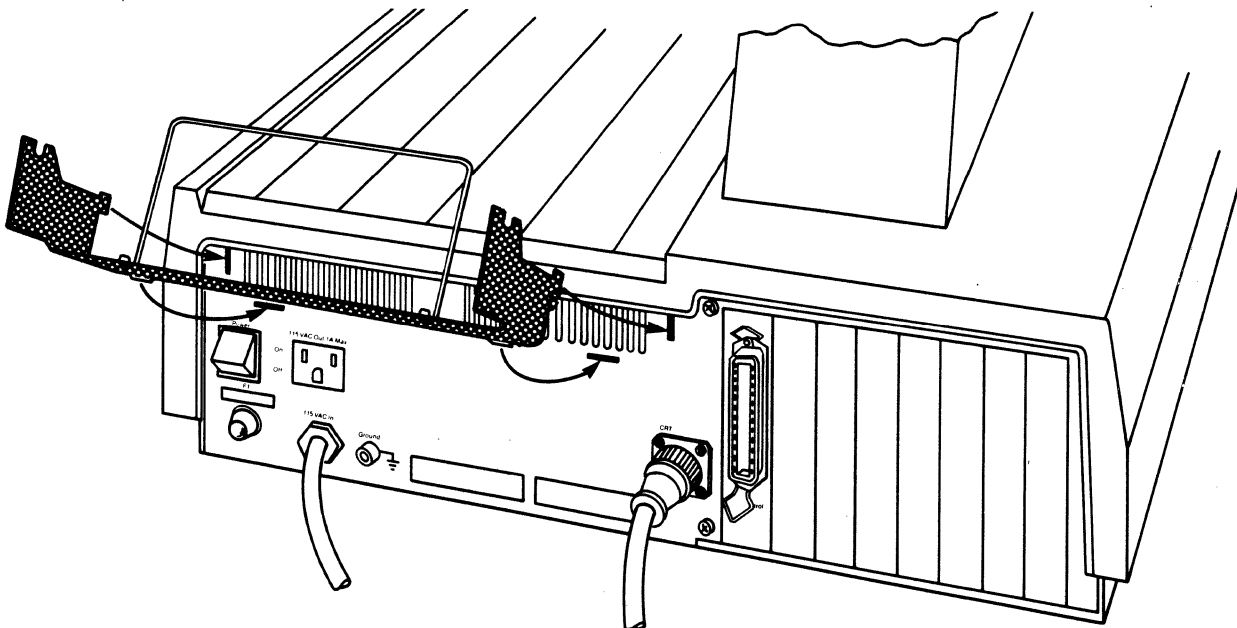
Figure 1-3. Rear View of Instrument with all Cables Properly Attached.

7. Thermal Paper Installation

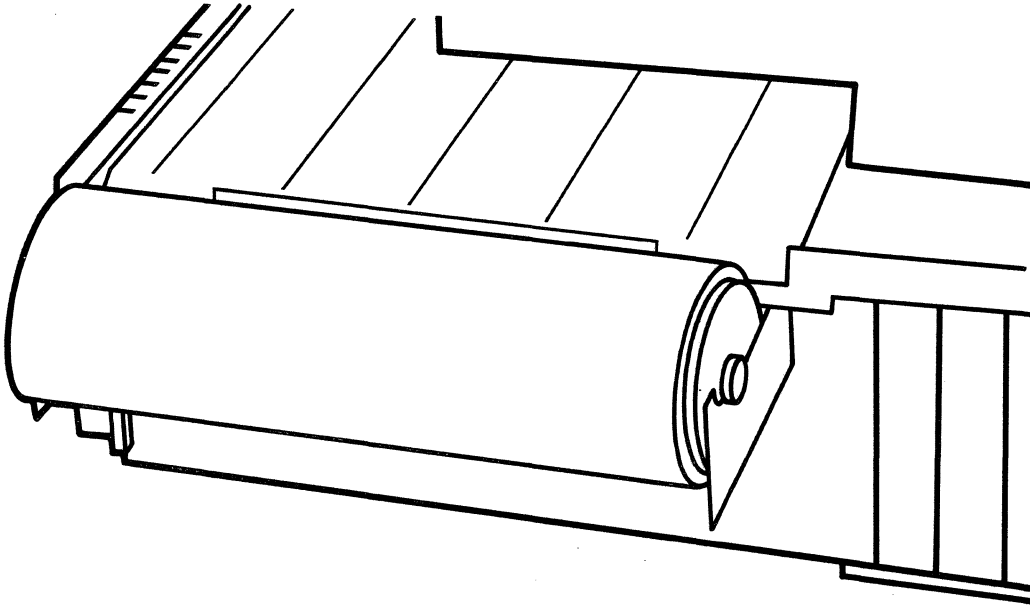
Installation of the thermal paper requires the following parts: the roll of thermal paper, two end caps, thermal paper holder and separator, as well as the thermal paper guide.



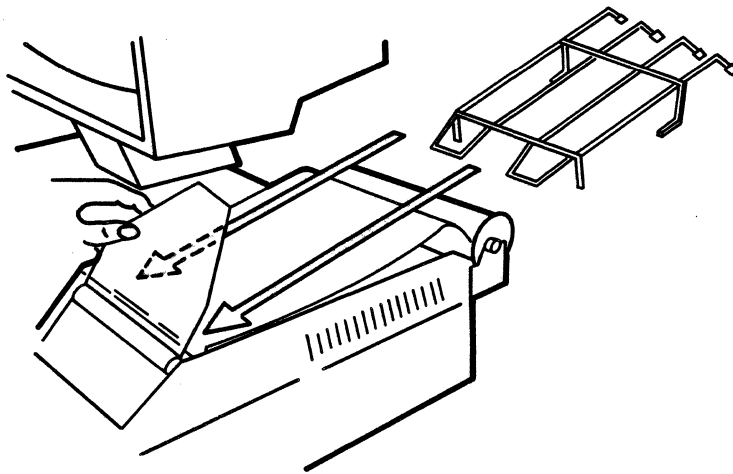
- a. Hold the thermal paper holder and separator with the rectangular wire loop facing up. Mount the paper holder by slipping the four tabs (two vertical and two horizontal) into the four slots on the back of the instrument.



-
- b. Insert one end cap into each end of the thermal paper roll. Place the roll in the semicircular notches of the paper holder.



- c. Pull the paper toward the front of the instrument with the blank side facing up. With the corners of the paper's leading edge folded in, lift the tinted plastic shield and feed the paper under and around the black roller. Ensure proper alignment of paper on both sides of the roller. This is accomplished by aligning the holes along the edges of the paper with the teeth in the paper advance rollers.



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- d. Install the paper guide over the chart paper so that the two straight legs fit into the two holes, and the two bent legs fit into the grooves.
 - e. Pull the paper over the guide and close the plastic shield.
8. The Optical Unit is equipped with two desiccant tubes at the bottom of its front side. These tubes are filled with silica gel beads at the factory to ensure a low humidity environment, and thus protect the optical components inside the unit.

Before attempting to operate the instrument, make sure that the silica gel is active, as indicated by the blue color of the desiccant beads. A pink color is an indication that the silica gel is ineffective and should be replaced. To change the desiccant, remove the tubes by pulling them straight out, unscrewing the top, and reloading with fresh silica gel.

Note: Exhausted desiccant can be regenerated by drying the beads at approximately 250°C overnight.

9. Press the operator's console switch to the ON position. Continue with the POWER-UP AND INITIALIZATION procedure described in Section 2.2.

2.0 OPERATION

2.1 INTRODUCTION

In this chapter the general operation of the 9420 and 9430 spectrophotometers is discussed. The three main points covered are: (a) Instrument Initialization; (b) Use of the Keypad; and (c) Selection of the Operating mode. A detailed operating procedure for each of the available methods is given in Sections 3 through 14.

2.2 POWER-UP AND INITIALIZATION

After the instrument has been unpacked, inspected and set up as described in Sections 1-6 and 1-7, the AC power cord for the Operator's Console is plugged into the utility outlet on the Optical Unit, and the console power switch is turned on. The Optical Unit power cord is plugged into a properly grounded receptacle. The power for the entire instrument can now be turned on and off using the power switch on the Optical Unit.

Open the sample compartment by sliding back the door on top of the compartment. Ensure that there are no samples placed in either the sample or reference cell holders. Close the sample compartment door, and turn on the instrument power.

The instrument will automatically begin an initialization procedure when the power is turned on. The initialization procedure consists of two parts; (1) a series of diagnostic routines and (2) a baseline correction program. The entire procedure takes approximately 17 minutes. The CRT displays a countdown of the time remaining in the initialization, as shown in Figure 2-1.

Note: The brightness control for the CRT display can be adjusted using the small screwdriver provided in the instrument tool kit. The brightness control is located on the back of the CRT, and is labelled "BRIGHT" (see Figure 1-3).

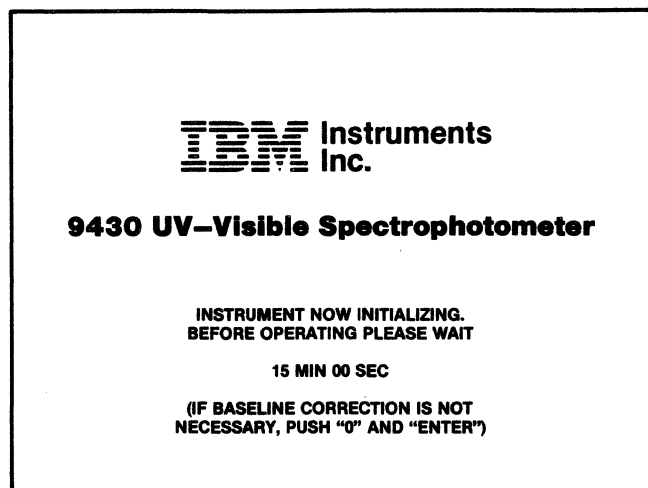


Figure 2-1. Initialization Display

If the baseline correction is not desired, that portion of the initialization can be skipped by pressing the following key sequence:

and where 0 = zero.

The initialization time will then be only 7 minutes. The message "BASELINE OFF" will appear on the CRT as a warning that no baseline correction was performed.

During the initialization procedure, tests are made of many of the mechanical components of the Optical Unit, including the filters and switchable mirrors. A number of clicks will be heard during these tests. These sounds are normal.

A number of the adjustments and calibrations made during the initialization procedure are temperature dependent. For very demanding work, it is recommended that the instrument be reinitialized after it has been allowed to warm up for 1 to 2 hours. This can be done by momentarily turning the power off, then turning it on again. This ensures that the instrument is initialized while all major components are at thermal equilibrium.

2.2.1 DIAGNOSTICS

During the first part of the initialization procedure, a number of diagnostic routines are performed. The major instrument components and functions that are tested include:

1. The random access memory (RAM).
2. Chopper mirror rotation.
3. Filter positioning.
4. The slit-drive control motor.
5. Source lamp operation.
6. Light source changeover.
7. The wavelength control mechanism.
8. The spectral bandwidth.
9. Wavelength calibration.
10. Wavelength reproducibility.
11. The initial operating conditions.

If any of the above items fails to meet specifications, an error message is displayed. A complete listing of the error messages, and the probable cause of each error are provided in Appendix A.

2.2.2 BASELINE CORRECTION

A baseline correction is performed during the second part of the initialization procedure. This procedure is used to flatten out any curvature that remains in the baseline after corrections are made for the change in source intensity as a function of wavelength. The baseline correction can also compensate for variations ($\pm 20\%$ T) in the light path between the sample and reference channels.

Baseline correction is performed under the following conditions:

- Spectral Bandwidth : 2 nm.
- Scan Speed : 100 nm/min.
- Time Constant : 0.4 sec.
- Sampling Interval : 1 point/nm.
- Wavelength range : 910 to 185 nm.

These conditions provide adequate baseline correction for most applications. The correction using these parameters can be repeated at any time using the baseline recorreption option available in Method 53. (See Section 12 for details of this procedure.)

For very precise measurements, it may be necessary to collect the baseline data under exactly the same conditions as the sample scan. This can be accomplished by using the real-time ratio and subtracting capabilities of Method 55. (See Section 14 for details of this procedure.)

2.3 THE KEYPAD

Once the power-up and initialization procedure is completed, all instrument functions are controlled from the keypad. The keypad is used to select the operating method, set the operating conditions and begin or stop a measurement. Figure 2-2 shows the position of the various keys on the keypad. Before beginning work with the instrument, it is recommended that the user become familiar with the locations and functions of the various keys as hereby provided.

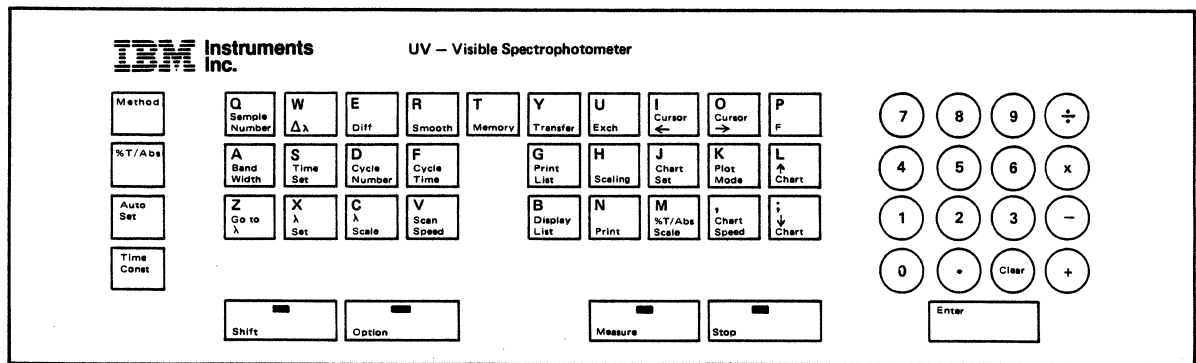


Figure 2-2. The 9420/9430 Keypad.

2.3.1 EXPERIMENTAL CONDITION KEYS

These keys are used to select the operating method, and to set the parameters for a given measurement. Table 2-1 summarizes the range of values allowed for each parameter, the standard value set for each parameter when the instrument is initialized and the methods in which each parameter is used. A brief description of each key is given below.

<u>Key</u>	<u>Function</u>
1. Method	Selection of operating method.
2. %T/ABS	Selection of photometric mode.
3. Time Const	Selection of filter time constant.
4. Bandwidth	Selection of spectral bandwidth. Note: For values less than 1.0, a leading zero must be entered (e.g., 0.2).
5. Go to λ	Sets instruments at a specified wavelength.
6. λ Scale	Sets: (1) Wavelength scale on printer/plotter. (2) Scan increment in Method 1 (see Table 3-1). (3) Scan increment in Method 5 (See Table 7-1).
7. Cycle Number	Sets number of measurement cycles.
8. Time Set	Sets the measurement time for a single cycle in Method 3. Note: When entering the time, separate the minutes and seconds with "-". Two digits must always be entered for the seconds (e.g., enter "001-05" for 1 minute, 5 seconds).
9. Cycle time	Sets the time from the beginning of one cycle to the beginning of the next. The time is entered as described above for t set.
10. Scan Speed	Sets the scanning speed during spectral measurement.
11. Chart Speed	Sets the speed of the chart paper in Method 3.
12. λ Set	Sets the wavelength region to be scanned. The upper limit must be entered first. Note: Maximum range is determined by λ Scale (see Table 3-1).

-
13. Used with λ Scale to set the wavelength interval for Method 5 (see Table 7-1).
 14. Sets the sample number.
Note: The sample number is automatically incremented after each measurement cycle.
 15. Sets the photometric range. The lower limit must be entered first.

2.3.2 PRINTER/DISPLAY CONTROL KEYS

This set of keys is used to control the printing and display of data on the printer/plotter or CRT. A description of each key is given below.

1. Prints a list of the parameters used for the measurement, on the thermal printer.
2. Prints the photometric value and wavelength currently displayed in the upper left corner of the CRT.
3. Sets the plotter to one of three modes; sequential, overlay, or off. The choices displayed are (1) SEQ, (2) OVERLAY and (3) OFF. Overlay plots can be made in Methods 1 and 11.
4. Used to select the line type for the plotter output. Selections are (1) FULL, (2) DOTTED, (3) BROKEN and (4) CHAIN.
5. Advances the chart paper.
6. Rewinds the chart paper.
7. Draws the axes on the plotter when overlay plots are made; can also be used to inhibit the drawing of axes. Choices are (1) ON, (2) OFF, (3) DRAW. In Method 4, this key is used to obtain a printout of the calibration curve data.
8. Displays the experimental parameters on the CRT. Pressing the key again returns the previously displayed data to the CRT.

Table 2-1. Parameters Used in Each Method

Function Key	Standard	Method*						Parameter Setting Selections
		#1	#2	#3	#4	#5	#6	
1. METHOD	1	X	X	X	X	X	X	0,1,2,3,4,5,6,11,51,52,53,54,55
2. %T ABS	1) % T	X	X	X		X	X	1) % T; 2)ABS
3. TIME CONST	0.4 sec	X	X	X	X	X	X	0.4,1,2,4 and 8 secs.
4. BAND WIDTH	2.00 nm	X	X	X	X	X	X	0.01 to 5.00 nm
5. GO TO	500.0 nm	X	X	X	X	X		910.0 to 185.0 nm
6. λ SCALE	20 nm/cm	X				X		0.5,1,2,5,10,20 and 40 nm/cm
7. CYCLE NO	1	X	X	X		X	X	1 to 100
8. t SET	999 min 0 sec			X				1 sec to 999 min. 59 sec.
9. CYCLE TIME	0 min 0 sec	X	X	X		X	X	1 sec to 999 min. 59 sec.
10. SCAN SPEED	100 nm/min	X				X		4,10,20,40,100,200, or 400 nm/min.
11. CHART SPEED	2 mm/min			X				2,5,10,20,50,100,200, or 400 mm/min
12. λ Set	900 - 200	X				X	X	910.0 to 185.0 nm
13. $\Delta\lambda$	1					X		1 - 9999
14. SAMPLE NUMBER	1		X		X			0 - 999
15. %T ABS SCALE	0 - 1 ABS 0 - 100%T	X		X			X	-5.000 to 5.000 ABS -500.0 to 500.0 %T

* Method refers to the methods in which the keys are used.

2.3.3 DATA HANDLING KEYS

These keys are used to perform various data handling functions in Method 11. A complete description of the command format for each key is given in Section 9. A brief description of each key is given below.

1. **Smooth** Used to smooth data stored in any memory location.
2. **Diff** Used to obtain a derivative of any stored spectrum.
3. **Memory** Used to specify a memory location for a stored spectrum. Location 0 to 4 can be specified. These are volatile memories. Data will remain in storage as long as the instrument power is on.
4. **Transfer** Used to move a stored spectrum from one memory location to another.
5. **Exch** Used to exchange the contents of two memory locations.
6. **Cursor ←** Used to move the cursor left. The position of the cursor will be displayed in the upper left corner of the CRT, and can be printed on the plotter using the Print key.
7. **Cursor →** Used to move the cursor right. The position of the cursor will be displayed in the upper left corner of the CRT, and can be printed on the plotter using the Print key.

2.3.4 SPECIAL FUNCTION KEYS

These keys are used to control the operation of the instrument.

1. **Measure** Used to start a measurement cycle. In Method 11, used to plot stored data.
2. **Stop** Used to interrupt a measurement cycle. Also used to cancel a parameter change if the Enter key has not been pressed. In Method 1, the Stop key can also be used to pause during a scan. (See Section 3.1.)
3. **Auto Set** The Auto Set key is used to set the baseline to 100.0%T or 0.000 ABS. The following procedure is used:

(a) Place the blank solution in both the sample and reference compartments.

(b) Press the **Auto Set** key. Two choices will be displayed - (1) ON and (2) OFF.

(c) Press **1** and **ENTER** to cause the Auto Set to begin. The reading will be set to 100.0% T or 0.000 ABS.

The key can only be used at a single wavelength. Each time the Auto Set key is used, the function is left in the "ON" state. During the baseline correction procedure, an Auto Set is performed at 500 nm. Therefore, immediately after power-up or baseline recorection, the Auto Set function will be "ON".

4. **Enter** The Enter key is pressed to indicate the end of a numeric entry or a command string.
5. **Clear** Used to clear an incorrect numeric entry. Clear can only be used if Enter has not yet been pressed.
6. **Shift**
Option
F These special keys are for use with optional accessories. Their use is described in the appropriate accessory manuals.

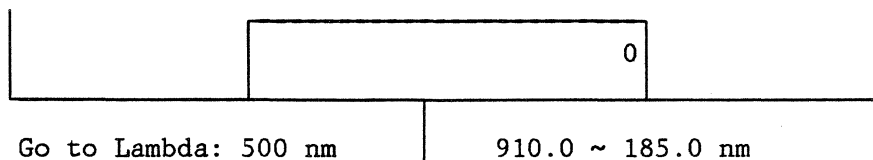
2.3.5 NUMERIC/ARITHMETIC KEYS

The keys 0-9, ÷, x, +, - and . are used to enter numeric values or select the appropriate arithmetic operation.

2.3.6 USING THE KEYPAD

The keypad responds with a single, audible "beep" when a key is pressed. If an invalid key is pressed, or an illegal entry is made, the instrument responds with three "beeps", indicating the error. Once a method is selected, keys not used for that method are disabled. The CRT is used to provide prompting during the selection of operating conditions. To change a parameter, the following procedure is used.

-
1. Press the function key for the operating condition that is to be changed. A prompt will appear at the bottom of the CRT, indicating the key that was pressed and the options that may be selected, as shown in the following example. If the **Go to λ** key is pressed, the following prompt appears on the CRT:



The value on the left indicates that the current wavelength setting is 500 nm. The values on the right show the valid range for this parameter, 910 - 185 nm.

2. Using the numeric keys, select a new value for the parameter. As the numeric keys are pressed, the new value will appear inside the box on the CRT. To complete the change, press the **Enter** key. The new value will then be added to the parameter list replacing the previous value.

The following rules summarize the common errors that can occur during use of the keypad.

1. If an unused key is pressed, the instrument will respond with three "beeps". The correct key can then be pressed.
2. If an unacceptable parameter is entered, three "beeps" will sound and the letter E will appear on the CRT next to the entry. Press **Clear** to remove the illegal entry from the screen and enter the correct value.
3. If the incorrect parameter key is pressed, the **Stop** key can be used to exit the parameter entry routine.
4. If an incorrect numeric key is pressed while entering a parameter change, but the **Enter** key has not yet been pressed, the **Clear** key can be used to clear the incorrect number.

2.4 METHOD SELECTION

Twelve operating methods have been programmed into the 9420 and 9430 spectrophotometers. In the sections that follow, each of these methods is

presented in a manner that will facilitate a thorough understanding of the instrument as rapidly as possible. A cursory explanation of these methods is presented below.

1. METHOD 0: TABLE OF CONTENTS

A listing of all of the available methods can be obtained on the CRT by entering METHOD 0. To enter METHOD 0, press the **Method**, **0** and **Enter** keys. The CRT listing is shown in Figure 2-3.

METHOD 0: TABLE OF CONTENTS
METHOD 0: TABLE OF CONTENTS
METHOD 1: SPECTRUM MODE
METHOD 2: FIXED WAVELENGTH MODE
METHOD 3: MEASUREMENT VERSUS TIME
METHOD 4: QUANTITATIVE ANALYSIS
METHOD 5: INCREMENTAL WAVELENGTH MODE
METHOD 6: WAVELENGTH PROGRAMMING
METHOD 11: SPECTRUM DATA PROCESSING
METHOD 51: SOURCE CONDITIONS
METHOD 52: CONTROL SETTINGS
METHOD 53: INITIALIZATION
METHOD 54: PMT VOLTAGE SETTING
METHOD 55: SPECTRAL SUBTRACT/RATIO MODE

Figure 2-3. Table of Contents Display, Method 0.

2. METHOD 1: SPECTRUM MODE

In Method 1, the transmittance or absorbance of a sample is measured as a function of wavelength. The spectrum can be displayed on either the CRT or the printer/plotter.

3. METHOD 2: FIXED WAVELENGTH MODE

This method is used to measure the change in absorbance or transmittance of a sample as a function of time. Each measurement is printed on both the CRT and the printer/plotter.

4. METHOD 3: MEASUREMENT VERSUS TIME

Method 3, like Method 2, is used to measure the change in absorbance or

transmittance of a sample as a function of time. In Method 3, however, the data is plotted as a strip-chart recording on the printer/plotter. A cursor marks the photometric value on the CRT.

5. METHOD 4: QUANTITATIVE ANALYSIS

In Method 4, a calibration curve can be generated for quantitative analysis of a single component in a sample. Both the calibration curve and the measured sample concentrations can be recorded on the printer/plotter.

6. METHOD 5: INCREMENTAL WAVELENGTH MODE

This method is used to measure transmittance or absorbance at fixed wavelength intervals across a given wavelength range. Each discrete measurement is printed on the printer/plotter.

7. METHOD 6: WAVELENGTH PROGRAMMING

Method 6 is used to measure the absorbance or transmittance of a sample at up to ten programmable wavelengths. A point plot of the data is drawn on the printer/plotter. Below each plotted point, the measured transmittance or absorbance is printed out.

8. METHOD 11: SPECTRUM DATA PROCESSING

In Method 11, a variety of data processing functions such as smoothing, differentiation and arithmetic manipulations can be performed on stored spectra.

9. METHOD 51: SOURCE CONDITIONS

This method is used to select the source operating conditions. The tungsten and deuterium lamps can be turned on and off manually, and the wavelength at which source changeover occurs can be selected (340.0-350.0 nm).

10. METHOD 52: CONTROL SETTINGS

Method 52 can be used to disable the audible tone produced by the keypad, to align the thermal head on the printer/plotter, and to select the number of digits used in the display of photometric measurements (4 digits or 5 digits).

11. METHOD 53: INITIALIZATION

This method is used to reset the instrument to the operating conditions

that are loaded after the power-up initialization routine. Method 53 can also be used to recorrect the baseline and to enter the date.

12. METHOD 54: PMT VOLTAGE SETTING

In Method 54, the instrument can be set to the single-beam mode at a fixed photomultiplier tube voltage.

13. METHOD 55: SPECTRAL SUBTRACT/RATIO MODE

Method 55 can be used to ratio or subtract spectra automatically during data acquisition.



3.0 METHOD 1 - TRANSMITTANCE OR ABSORBANCE SPECTRUM

3.1 DESCRIPTION

Method 1 is used to measure the absorbance or transmittance spectrum of a sample. Considerable flexibility is provided in setting the data collection parameters, and in the display of the resulting spectrum. In this section, the use of Method 1 is described in detail.

The parameter keys used in Method 1 are described briefly in Section 2.3. The following keys merit additional comment.

1. **%T/ABS** Scale is used to set the photometric scale for the real-time display of data on the CRT, and the on-line plotting of data on the printer/plotter. If the spectrum goes offscale it is not necessary to recollect the data. The spectrum is automatically stored in memory, and can be replotted on the correct scale using Method 11, as described in Sections 9.3 and 9.4.
2. **λ Scale** serves two functions in Method 1. (1) It is used to set the wavelength axis scale for printer/plotter output of spectra, and (2) it is used to set the wavelength increment between successive data points in the scan. The scan increments that result for each choice of λ scale are shown in Table 3-1, and are given by equation 3-1.

$$\text{Scan Increment} = \lambda \text{ scale} / 50. \quad (3-1)$$

Table 3-1. Interdependence of Scan Parameters.

λ Scale (mm/cm)	Scan Speed (nm/min)	Scan Range (nm)	Scan Increment (nm)
0.5	4 - 10	0.5 - 20	0.01
1.0	4 - 20	1.0 - 40	0.02
2.0	4 - 40	2.0 - 81	0.04
5.0	4 - 100	5.0 - 204	0.10
10	4 - 200	10 - 408	0.20
20	4 - 400	20 - 725	0.40
40	4 - 400	40 - 725	0.80

-
3. Both the Scan Range, determined by λ set, and the Scan Speed are dependent on the value selected for λ scale. This is done to prevent collection of data files that contain more than 2040 data points, and to avoid exceeding the maximum data collection rate of the instrument.

If an improper choice of parameters is made two error messages are displayed, when the MEASURE or GO TO λ keys are pressed;

E020: This message indicates that the λ Scale and λ Set values are incompatible.

E021: This message indicates that the λ Scale and Scan Speed are incompatible.

If either of these errors is encountered, the following steps should be taken:

- a. Press the Stop key to clear the error state.
 - b. Select compatible values for the appropriate parameters using the values listed in Table 3-1.
4. The Chart Set, Scaling and Plot Mode keys are used to control the printer/plotter. Chart Set is used to select the operating mode of the printer/plotter. Three choices are available, (1) SEQ, (2) OVERLAY and (3) OFF. In the SEQ mode, spectra are plotted sequentially as they are collected. One spectrum will be drawn on each set of axes.

In the OVERLAY Mode, several spectra can be drawn on a single set of axes. After each plot, the printer/plotter will rewind to the beginning of the spectrum.

In the OFF mode, the printer/plotter is disabled. This stops the automatic plotting of data during collection. The data can still be plotted, off-line by using Method 11 (see Section 9.4).

Scaling controls the drawing of the axes for the plot. If Scaling is set to (1) ON, the axes will automatically be drawn for each plot. This mode is usually selected for sequential plotting.

If overlay plots are made, the (3) DRAW mode can be selected. The axes are drawn once, and the scaling function is automatically set to (2) OFF. This inhibits the drawing of the axes after each spectrum in the overlay plot.

Plot Mode is used to select the line type for the plot; full, dotted, broken or chain.

Instruction for using Chart Set, Scaling and Plot Mode are given in the procedure section which follows.

5. The **Cycle No** and **Cycle Time** keys can be used to automatically repeat a scan at preset time intervals. When multiple cycles are run, the successive spectra will be overlaid on the CRT.
6. The **STOP** key can be used to pause during a scan, or to abort a scan. If the stop key is pressed once during a scan, the scan will pause at the current wavelength.

The red light at the top of the **STOP** key will flash, indicating the pause state. If the plotter and CRT are lagging behind the actual scan, the display will be updated to the wavelength at which the stop key was pressed. To resume the scan, the **Measure** key is pressed. To abort the scan, the **STOP** key is pressed a second time.

3.2 PROCEDURE

1. Press the **Method**, **1** and **Enter** keys. A grid for the display of spectral data will appear on the CRT, as shown in Figure 3-1.

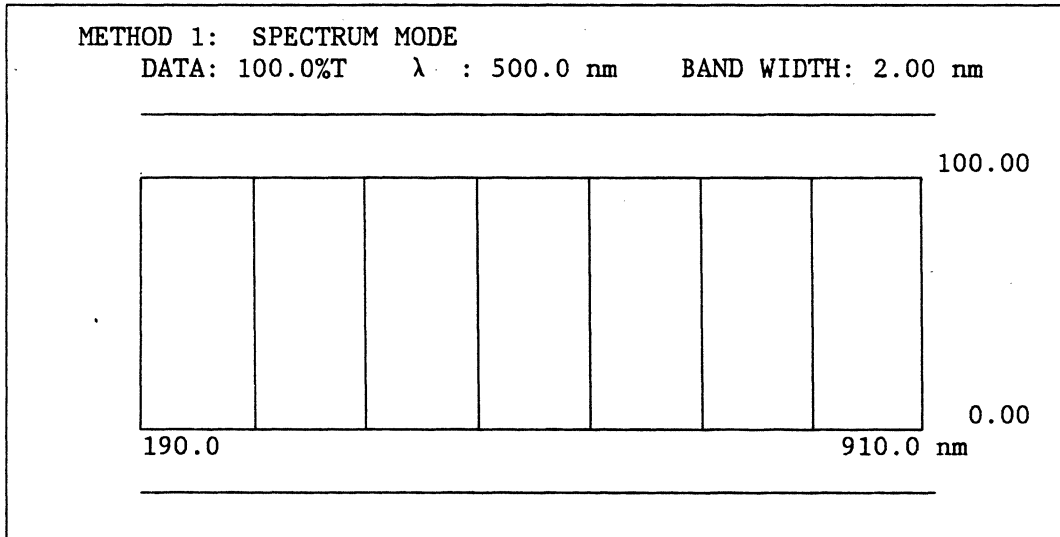


Figure 3-1. Method 1 Display

-
2. To check the operating conditions, press **Display List**. The preset operating conditions will appear on the CRT as shown in Figure 3-2.

METHOD 1: SPECTRUM MODE	
Data: 100.0%T : λ : 500.0 nm BAND WIDTH: 2.00 nm	
<hr/>	
TIME CONST	: 0.4 sec
AUTO SET	: (1) ON
λ SET	: 900.0 ~ 200.0 nm
λ SCALE	: 20 nm/min
SCAN SPEED	: 100 nm/min
%T ABS SCALE	: 0.0 ~ 100.0%T
CYCLE NO.	: 1
CYCLE TIME	: 0 min 0 sec
SCALING	: (1) ON
CHART SET	: (1) SEQ
PLOT MODE	: (1) FULL
<hr/>	
TIME CONST.	: 0.4 sec 0.4, 1, 2, 4 8 sec

Figure 3-2. Method 1 Operating Conditions

3. To change any parameter, press the appropriate parameter key. For example, to change the value of the time constant, press the **Time Const** key. The current value of the parameter is displayed at the bottom of the CRT, together with a list of the allowed values, as shown in Figure 3-2. A new value can be entered using the numeric keypad, and **Enter**.
4. If an automatic plot of the spectrum is desired, the following parameters should be set:
- Use the **Chart Set** key to select either (1), sequential, or (2), overlay plotting.
 - Set the **Scaling** key to (1), ON, to provide automatic generation of the axes for each plot. In overlay mode, the axes will be drawn for the first plot only. Alternatively, the (3) Draw function can be used to draw the axes immediately. The status will change to (2) OFF after the axes are drawn.

-
- c. Use the **Plot Mode** key to select the line type for the plot, (1) FULL, (2) DOTTED, (3) BROKEN or (4) CHAIN. The plot mode can be changed between successive scans.
 5. If no plotting is required, select the **Chart Set** option (3), OFF.
 6. When all the parameters have been selected, press **Display List** again, to return the display to the measurement grid, as shown in Figure 3-1.
 7. To produce a header for the spectrum on the printer/plotter, and to obtain a list of the parameters used for the scan, press **Print List**. A sample listing is shown at the top of Figure 3-3. The date will appear on the first line of the header, if the date was entered using Method 53 (see Section 12.2).
 8. The **Auto Set** key can be used to set the baseline to 100.0 %T or 0.000 ABS at the starting wavelength of the scan. If the monochromator is not set to the correct starting wavelength, the **Go To λ** key can be used to correct the setting.
 9. Place the sample in the sample chamber, close the door and press **MEASURE**. The light on the measure key will come on, indicating that the measurement is in progress. As the spectrophotometer acquires the photometric data, the curve will be displayed on the CRT. Concurrently, the value of each plotted point is numerically displayed, in the appropriate units, in the upper left corner of the CRT.
 10. To pause during a scan, press the **STOP** key once. To continue the scan press **MEASURE**. To abort the scan, press **STOP** twice.
 11. If an automatic plot was selected, a spectrum will be generated, as shown in Figure 3-3.
 12. When the scan is completed, the data is automatically stored in memory 1. Further data manipulations can be performed using Method 11. Details are provided in Section 9.0, Data Processing of Spectra.

DATE: 07,01,83 OPERATOR: _____ SAMPLE: _____
CELL: _____ TEMPERATURE: _____ SOLVENT: _____
HUMIDITY: _____ CONC: _____

DATA MODE : (2) ABS
BAND WIDTH : 1.00 nm
TIME CONST : 0.4 sec
 λ SET : 500.0 ~ 400.0
 λ SCALE : 10 nm/cm
SCAN SPEED : 100 nm/min
%T ABS SCALE : 0.000 ~ 1.800 ABS
CYCLE NO : 1

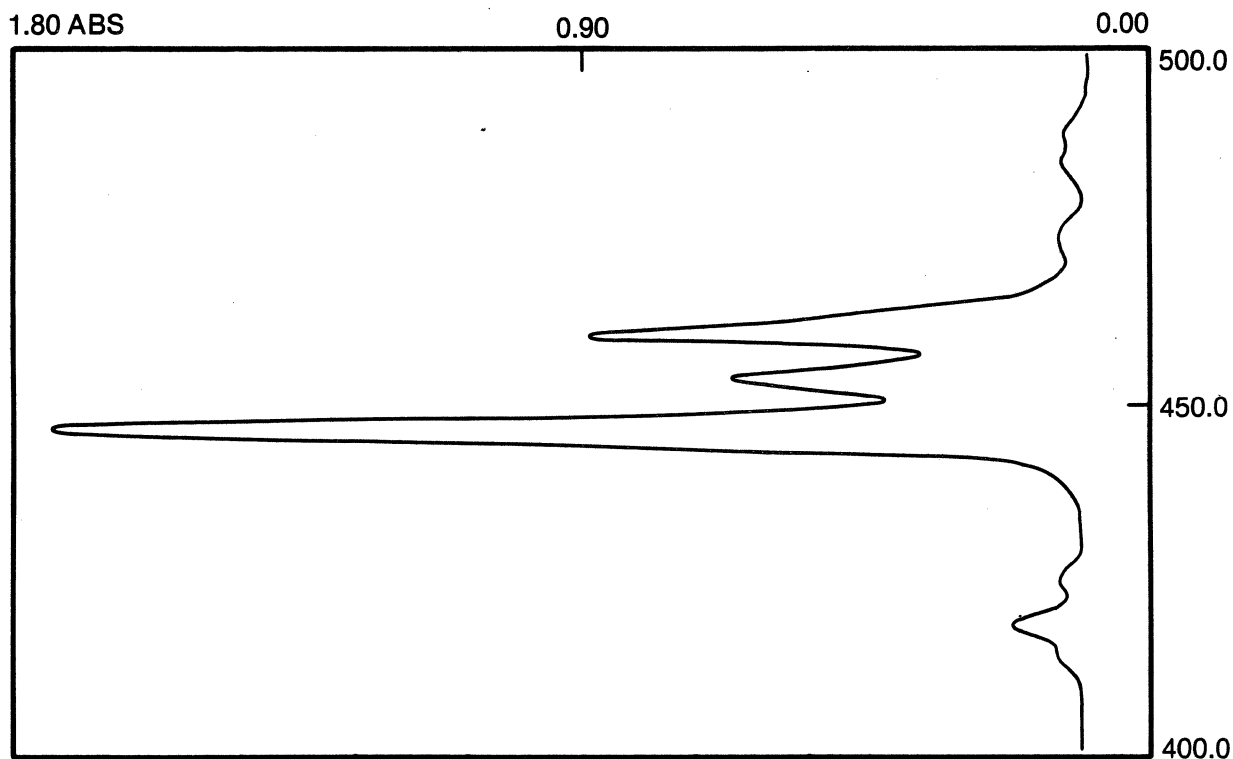


Figure 3-3. Spectral data plotted using Method 1.
Display List was used to record the operating conditions.

4.0 METHOD 2 - TRANSMITTANCE OR ABSORBANCE VERSUS TIME AT A FIXED WAVELENGTH (PRINTOUT)

4.1 DESCRIPTION

This method is intended for monitoring the change in a sample's absorbance or transmittance as a function of time. Both the time between measurements and the number of measurements to be made can be selected. The results are displayed on the CRT, and printed on the printer/plotter.

4.2 PROCEDURE

Perform the following steps:

1. Press the **Method**, **2** and **Enter** keys. The CRT display will appear as shown in Figure 4-1.

```
METHOD 2:  FIXED WAVELENGTH MODE
DATA: 100.0%T   λ: 500.0 nm   BAND WIDTH: 2.00 nm
-----
SAMPLE NO      DATA      MODE      λ(nm)  CYCLE NO.
-----
```

Figure 4-1. Method 2 Display

-
2. To check the operating conditions, press the **Display List** key. The current values of the operating parameters will be displayed as shown in Figure 4-2.

```
METHOD 2:  FIXED WAVELENGTH MODE
DATA: 100.0%T   λ: 500.0 nm   BAND WIDTH: 2.00 nm

-----

TIME CONST   : 0.4 sec.
AUTO SET     : (1) PREVIOUS
GO TO λ      : 500.0 nm.
CYCLE NO     : 1
CYCLE TIME   : 0 min 0 sec
CHART        : (1) ON

-----
```

Figure 4-2. Method 2 Operating Conditions.

3. Select the desired measurement wavelength by pressing the **Go To λ** key and entering the appropriate numeric value. The **Auto Set** key can be used to set the baseline to 0 ABS or 100%T, if necessary.
4. The number of measurements to be made is selected using the **Cycle No** key.
5. Select the delay between measurements using the **Cycle Time** key. The time is entered as minutes and seconds. For example, to select a cycle time of 1 minute 30 seconds, enter "001-30". The hyphen ("-") is used to separate the minutes and seconds. If a cycle time of zero is selected (preset condition), the instrument will cycle at its maximum rate, approximately one measurement every 0.7 seconds.

NOTE: At the fastest cycle times (0 or 1 second), the thermal printer can cause the measurement time to increase. This can be avoided by using the **Chart Set** key to disable the printer (see Section 3.2.6), and by observing the data as displayed on the CRT.
6. Place the sample in the sample chamber, and enter the sample number by pressing **Sample Number**, the appropriate numeric keys and Enter.

-
7. Press the Measure key. The measurements will begin immediately. The data will be presented on the CRT and printed on the printer/plotter. Figure 4-3 shows a sample of the printer/plotter output obtained. Four samples were run, with three measurement cycles per sample. If a hard copy is not needed, the **Chart Set** key is used to disable the printer.

SAMPLE NO.	DATA MODE	λ nm	CYCLE NO.
1	96.1 %T	900.0 nm	1
	96.5 %T	900.0 nm	2
	96.3 %T	900.0 nm	3
2	6.0 %T	900.0 nm	1
	6.0 %T	900.0 nm	2
	6.0 %T	900.0 nm	3
3	77.9 %T	900.0 nm	1
	77.7 %T	900.0 nm	2
	77.4 %T	900.0 nm	3
4	31.4 %T	900.0 nm	1
	31.1 %T	900.0 nm	2
	31.2 %T	900.0 nm	3

Figure 4-3. Sample output for Method 2.
Four samples, with a cycle number = 3 are shown.



5.0 METHOD 3 - TRANSMITTANCE OR ABSORBANCE VERSUS TIME AT A FIXED WAVELENGTH (PLOTTED)

5.1 DESCRIPTION

This method is similar to Method 2. It also measures either absorbance or transmittance as a function of time. The results of the test are plotted on the printer/plotter. The output is a stripchart recording of the photometric value as a function of time. In this mode the instrument can be used to monitor a reaction, or as a detector for a chromatographic system (with the appropriate flow cell accessories).

5.2 PROCEDURE

1. Press the **Method**, **3** and **Enter** keys to enter the method. The CRT will display the measurement axis as shown in Figure 5-1.

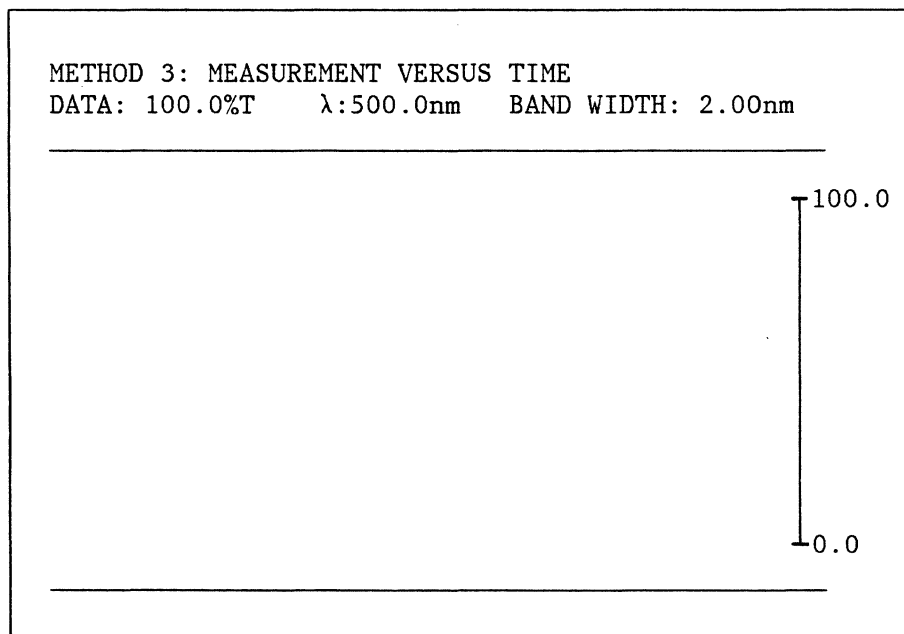


Figure 5-1. Method 3 Display

2. If a check of the operating conditions is desired, press Display List. The parameters used to set the operating conditions will be displayed as shown in Figure 5-2.

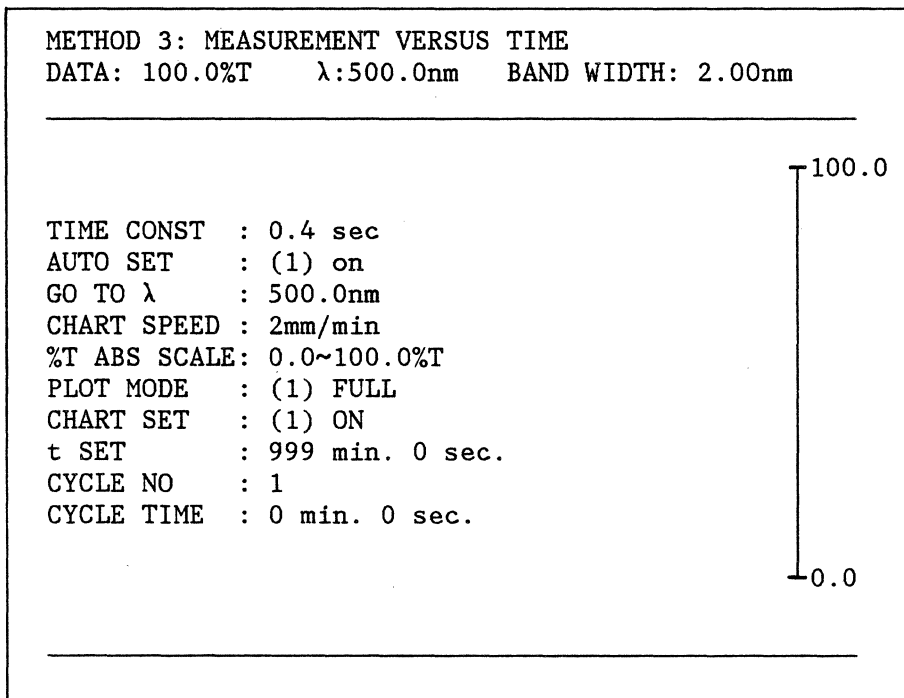


Figure 5-2. Method 3 Operating Conditions

3. To set the measurement wavelength, use the **Go To λ** key. If necessary, the **Auto Set** key can be used to set the baseline to 100.0%T or 0.000 ABS at the selected wavelength.
4. To set the measurement time, use the **t Set** key. The time should be entered as minutes and seconds, separated by a hyphen (-). For example, to set the time to 1 minute and 5 seconds, enter "001 - 05". Two digits must always be entered for the seconds.
5. The **Cycle Number** and **Cycle Time** keys can be used to automatically repeat the measurement cycle. The cycle time is measured from the beginning of one cycle to the beginning of the next. Therefore, the cycle time should be greater than the measurement time (Cycle Time > t Set). The photometric axis is not redrawn automatically at the beginning of each cycle, so the cycles appear as a single, continuous strip-chart recording.

-
6. When all the parameters have been entered, press **Print List** to obtain a heading for the data, as shown at the top of Figure 5-3.
 7. Press **SCALING** to draw the photometric axis on the printer/plotter. The axis will be drawn immediately when the key is pressed.
 8. Place the sample in the sample chamber, and press **Measure** to begin the experiment. The chart paper will begin to advance at the rate determined by the **Chart Speed** key. A strip-chart recording of the absorbance or transmittance of the sample will be drawn on the printer/plotter. During the measurement, a cross will appear on the CRT, marking the absorbance or transmittance of the sample on the photometric axis.
 9. The measurement will stop automatically at the end of the measurement time (t SET), or at the end of the last cycle. The experiment can be terminated at any time by pressing the **Stop** key.
 10. A sample of the printer/plotter output for Method 3 is shown in Figure 5-3.

DATE: __, __, __ OPERATOR: _____ SAMPLE: _____
CELL: _____ TEMPERATURE: _____ SOLVENT: _____
HUMIDITY: _____ CONC: _____

DATA MODE : (2) ABS
BANDWIDTH : 0.50 nm
TIME CONST : 0.4 sec
GO TO λ : 351.2 nm
t SET : 15' 0"
CHART SPEED : 10 mm/min
%T ABS SCALE : 0.000 ~ 1.500 ABS
CYCLE NO : 1
CYCLE TIME : 1' 0"

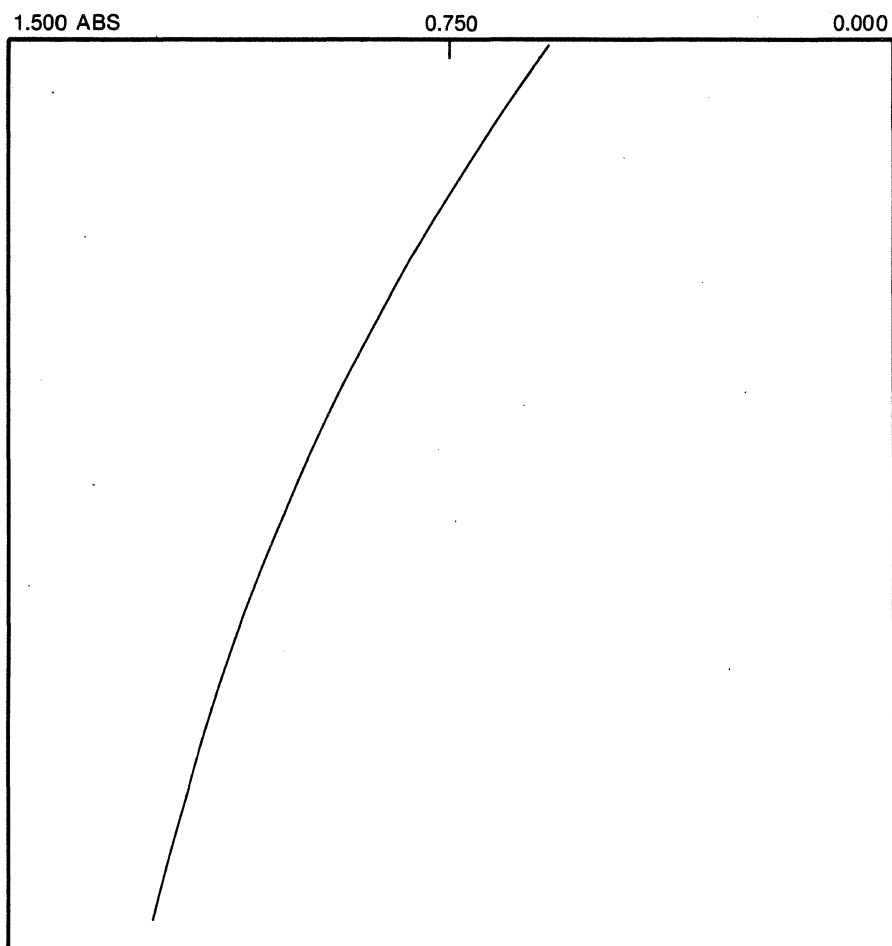


Figure 5-3. Sample Output for Method 3.

6.0 METHOD 4 - QUANTITATIVE ANALYSIS

6.1 GENERAL INFORMATION

6.1.1 DESCRIPTION

Beer's Law states that the absorbance of a sample is directly proportional to the concentration of the absorbing species. This relationship forms the basis for using spectrophotometry in quantitative analysis.

The 9420/30 quantitative analysis package is implemented in Method 4. This package can handle a variety of analytical situations, including those in which deviations from Beer's Law occur. A convenient, menu-driven format is used to guide the operator through the various phases of the analysis:

- A. Wavelength Selection.
- B. Measuring the Blank.
- C. Entering the Standard Concentrations.
- D. Measuring or Entering the Absorbance of the Standards.
- E. Selecting the Calibration Curve.
- F. Measuring the Unknown Sample Concentration.

This menu offers considerable flexibility. Three methods are available for defining the baseline (at the measurement wavelength) in Phase A. Up to 20 standards can be used to define the calibration curve. Absorbance values for the standards can either be measured directly, or entered from the keypad in Phase D. Three types of calibration curves can be used to calculate the sample concentration. The selected calibration curve is displayed on the CRT, and can be plotted on the printer/plotter, together with all of the conditions used for the analysis. Two commands, "+ Enter" and "- ENTER" are used to move back and forth through the menu easily. A complete description of each step in the menu is given in the sections that follow.

6.1.2 PROCEDURE

1. Before entering Method 4, select the bandwidth and time constant desired for the measurement. This can conveniently be done in Method 1. If necessary, use the Auto Set key to set the baseline to 0.000 ABS. These keys can also be accessed from Phase F of the Menu while in Method 4.

2. Press **Method** **4** and **Enter** to enter the menu. The menu is divided into 3 pages of CRT display. The first page covers Phases A through D, as shown in Figure 6-1. The second page covers Phase E and displays the calibration curve. The last page displays the measured absorbance and the calculated concentration as each sample is measured.
3. A blinking cursor will appear in Phase A of the menu.

```

METHOD 4: QUANTITATIVE ANALYSIS
DATA: 0.000 ABS   λ 500.0 nm   BAND WIDTH: 2.00 nm
-----
      OPERATE AS FOLLOWS:

A:  λ SET

■  λ0:500.0 nm   λ1:      nm   λ2:      nm

B:  SET BLANK, and PUSH "MEASURE"
    BLANK: 0.000 ABS

C:  INPUT CONC VALUES OF THE STANDARD SAMPLES (≤20)

    1:          2:          3:          4:          5:
    6:          7:          8:          9:          10:
    11:         12:         13:         14:         15:
    16:         17:         18:         19:         20:

D:  PUSH "MEASURE" FOR THE SAMPLE OR "ENTER" FOR DATA

    1:          2:          3:          4:          5:
    6:          7:          8:          9:          10:
    11:         12:         13:         14:         15:
    16:         17:         18:         19:         20:

    PUSH "+", "ENTER", CONTINUE TO NEXT PAGE

-----
                    ■ 0
  
```

Figure 6-1. Method 4 Display - Page 1.
 ■ Blinking Cursor

6.2 PHASE A - WAVELENGTH SELECTION

6.2.1 DESCRIPTION

One, two or three wavelengths can be used to measure the absorbance of the sample. These wavelengths are indicated as λ_0 , λ_1 and λ_2 in the menu. The absorbances measured at these three wavelengths will be referred to as A_0 , A_1 and A_2 , respectively.

Figure 6-2 illustrates the three procedures used to measure the sample absorbance, ΔABS . The appropriate procedure is selected by inspection of the sample spectrum, which can be obtained using Method 1. The three procedures are described below.

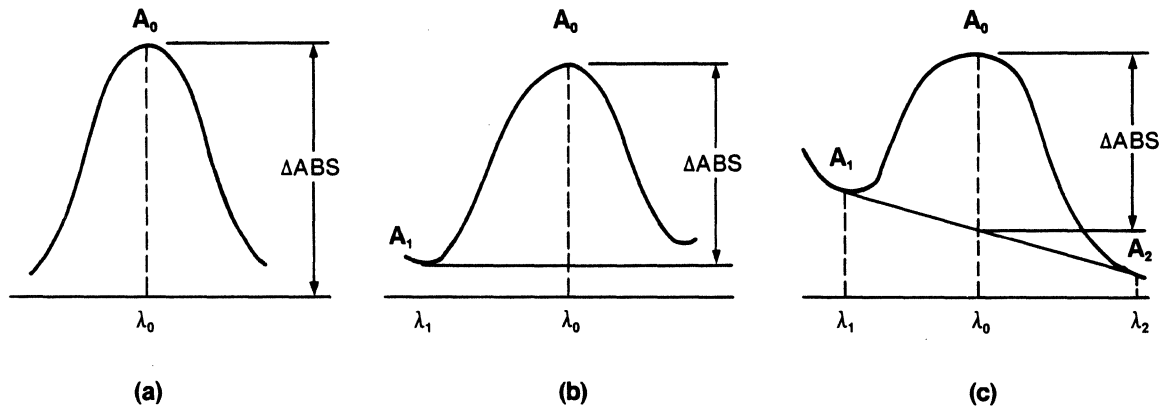


Figure 6-2. Procedures Used to Measure the Sample Absorbance (ΔABS).

1. In Procedure 1, one wavelength (λ_0) is used. The absorbance at λ_0 is measured. In this case $\Delta\text{ABS} = A_0$. This procedure should be used when the spectral baseline is flat and near zero, as shown in Figure 6-2(a).

-
2. In Procedure 2, λ_0 and λ_1 are used. The absorbance at λ_1 is subtracted from the absorbance measured at λ_0 . This method is useful when the baseline is nearly flat, but non-zero (see Figure 6-2(b)). $\Delta\text{ABS} = A_0 - A_1$.
 3. In Procedure 3, λ_0 , λ_1 and λ_2 are used. A tangent is drawn between λ_1 and λ_2 . ΔABS is calculated as shown in Figure 6-2. This method can be used when the baseline is sloped as shown in Figure 6-2(c). In this case, ΔABS is given by the following equation:

$$\Delta\text{ABS} = A_0 - \frac{|\lambda_1 - \lambda_0|A_2 + |\lambda_2 - \lambda_0|A_1}{|\lambda_2 - \lambda_1|}$$

6.2.2 PROCEDURE

1. If the one-wavelength method is being used, enter λ_0 by pressing the appropriate numeric keys and **Enter**. Press **+** and **Enter** to move to Phase B.
2. If the two-wavelength method is to be used, enter the values of both λ_0 and λ_1 , then press **+** and **Enter** to move to Phase B.
3. If the three-wavelength method is used, enter λ_0 , λ_1 , and λ_2 . The cursor will then automatically move to Phase B.

6.3 PHASE B - MEASURING THE BLANK

6.3.1 DESCRIPTION

This step measures the absorbance of the blank solution. Typically, the blank solution contains the solvent used to dissolve the sample, and any reagents used for the analysis. The absorbance of this blank solution, in the absence of analyte, is then subtracted automatically from all subsequent measurements.

6.3.2 PROCEDURE

1. Place the appropriate solution in the reference cell holder. Typically, the reference solution will be the solvent used for the analysis.
2. Place the blank solution into the sample cell holder and press **Measure**. The instrument will measure the blank absorbance using the procedure defined in Phase A, and display the value measured on the CRT. The cursor will automatically move to Phase C of the menu.
3. The last measured blank absorbance is always displayed on the CRT. To retain this value, simply press the **+** and **Enter** keys. The cursor will move to Phase C of the Menu.

6.4 PHASE C - ENTERING THE STANDARD CONCENTRATIONS

6.4.1 DESCRIPTION

In this step, the concentration of each standard is entered. The concentrations should be entered in the order in which the standards will be measured. These concentrations will be used, together with the absorbance of each standard, to construct the calibration curve.

6.4.2 PROCEDURE

1. To enter a new concentration, press the appropriate numeric keys and **Enter**. The cursor will automatically move to the next concentration value. Up to 20 concentrations can be entered.
2. When the last concentration has been entered, press the **+** and **Enter** keys. The remaining standard concentrations will be cleared, and the cursor will move to Phase D of the menu. For example, if five concentrations are entered followed by **+** and **Enter**, values 6 through 20 will automatically be cleared, and the cursor will move to Phase D.
3. If, after leaving Phase C, an error is noticed in one of the values press the **-** and **Enter** keys to return to step C. Press **Enter** repeatedly until the cursor is positioned in front of the value to be changed. Enter the

correct value. Use the **Enter** key to move past the last data point, then use the **+** and **Enter** keys to advance to Phase D.

4. The last entered concentration values will be displayed on the CRT. To retain these values, press the **+** and **Enter** keys, and the cursor will move to Phase D of the menu.

6.5 PHASE D - MEASURING OR ENTERING THE ABSORBANCE OF THE STANDARDS

6.5.1 DESCRIPTION

The absorbances of the standard solutions are measured in this step. If the values are already known, they can be entered from the keypad.

6.5.2 PROCEDURE

1. Place the standard corresponding to the first entered concentration into the sample holder. Press **Measure**. The absorbance will be measured using the wavelength(s) specified in Phase A. The absorbance of the blank will be subtracted out, and the result will be displayed on the CRT. The cursor will automatically move to the next position.
2. Repeat this process until all the standards have been measured. It is not possible to exit from Phase D until one absorbance value has been entered for each concentration value.
3. If the absorbances of the standards are known from a previous measurement, they can be entered from the keypad. In this case, press the appropriate numeric keys and **Enter**.
4. When the last value has been entered, press the **+** and **Enter** keys to proceed to Phase E.

6.6 PHASE E - SELECTING THE CALIBRATION CURVE

6.6.1 DESCRIPTION

In this phase, one of three methods is selected to establish the calibration curve that will be used to calculate the concentration of the sample. The method selected depends on the shape of the calibration curve itself.

When Phase E is entered, the second page of the Menu is displayed on the CRT. The data points collected for the calibration curve are plotted automatically, as shown in Figure 6-3.

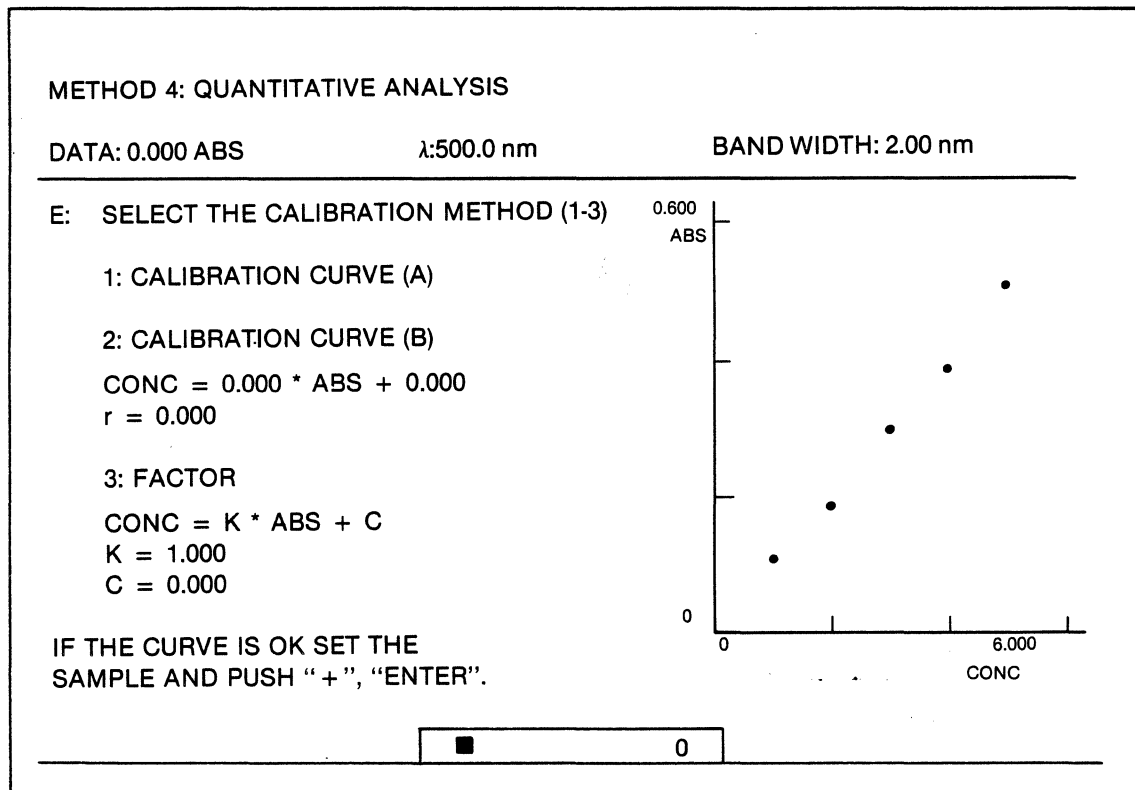


Figure 6-3. Calibration Curve Selection

For most analytical work, the plot of absorbance versus concentration will be linear, in accordance with Beer's Law. In these cases, METHOD 2: CALIBRATION CURVE (B) or METHOD 3: FACTOR should be used. If, however, the plot is non-linear, METHOD 1: CALIBRATION CURVE (A) may be used. The three methods are defined below.

1. CALIBRATION CURVE (A). In this method, a curvilinear approximation is made to the calibration curve. The data points are connected with straight line segments, and the sample concentration is calculated by interpolation. This method should only be used if the standard absorbances change monotonically with increasing concentration.
2. CALIBRATION CURVE (B). This method uses a least-squares algorithm to calculate the best straight line through the data points. The slope and intercept of the line are displayed on the CRT. A value for the correlation coefficient (r) is also displayed. A value of 1.0 indicates that the data is perfectly linear.
3. FACTOR. This method allows the slope and intercept for a linear calibration curve to be entered manually. If this method is used, the calibration curve data in Phases C and D are ignored. No standards are needed if this method is used.

6.6.2 PROCEDURE

1. The data will be displayed automatically on the CRT, as shown in Figure 6-3. After examining the point plot, select Option 1, 2, or 3.
2. If Option 1 is selected, the data points will be connected by straight line segments in order of increasing absorbance. No extrapolation will be made beyond the limits of the data points. The cursor will move to the bottom of the CRT. If the resulting plot is acceptable, press the + and Enter keys to proceed to Phase F. If it is not acceptable, use the - and Enter keys to step back in the Menu for the purpose of selecting another option.
3. If Option 2 is selected, the best straight line will be drawn through the data points. The line will be extrapolated beyond the limits of the data points. The calculated slope, intercept and correlation coefficient for the line are displayed. If the plot is acceptable, press the + and Enter keys.

-
- If Option 3 is selected, the cursor will move to the line labelled "K =". Enter the slope of the desired line. The cursor will then move to the next line, labelled "C =". Enter the intercept of the line. When both values have been entered press the + and Enter keys.

6.7 PHASE F - MEASURING THE UNKNOWN SAMPLE

6.7.1 DESCRIPTION

In this step, the absorbance of the sample is measured, using the wavelengths specified in Phase A. The absorbance of the blank, measured in Phase B, is subtracted out, and the result is displayed on the CRT. This absorbance is used to calculate the sample concentration by use of the calibration curve. The results are automatically displayed on the CRT and printed on the printer/plotter.

During this step, the third and final page of the Menu is displayed on the CRT, as shown in Figure 6-4.

METHOD 4: QUANTITATIVE ANALYSIS		
DATA: 0.000 ABS	λ: 500.0 nm	BAND WIDTH: 2.00 nm
SAMPLE NO.	ABS	CONC
1	0.254 ABS	2.540 CONC
2	0.743 ABS	7.430 CONC

Figure 6-4. Phase F - Measurement of the Unknown Samples.

At this time a number of keys become active. These keys are listed below.

- Method** - The **Method** key can be used to exit the quantitative analysis program and to enter another method.

DATE: __, __, __ OPERATOR: _____ SAMPLE: _____
 CELL: _____ TEMPERATURE: _____ SOLVENT: _____
 HUMIDITY: _____ CONC: _____

BAND WIDTH : 0.50 nm
 TIME CONST : 0.4 sec

A: λ SET λ_0 : 500.0 nm

B: BLANK ABS: 0.001 ABS

C: CONCENTRATION VALUE OF THE STANDARD SAMPLE

1: 1.000 2: 2.000 3: 3.000 4: 4.000 5: 5.000

D: 1: 0.110 2: 0.190 3: 0.300 4: 0.390 5: 0.510

λ_0 λ_0 λ_0 λ_0 λ_0

E: WAY OF ANALYSIS

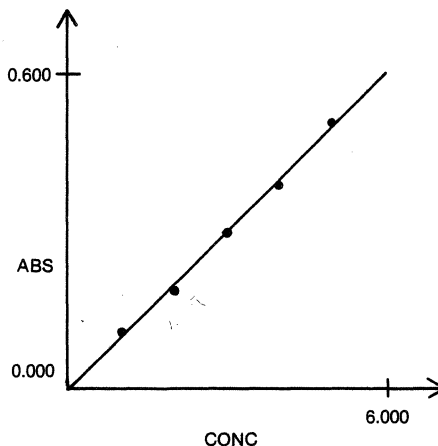
2: CALIBRATION CURVE B

CONC = K * ABS + C

K = 9.960

C = 0.012

r = 0.998



S. No.	λ_0	λ_1	λ_2
1	0.075 ABS 0.759 CONC	0.076 ABS	ABS ABS
2	0.319 ABS 3.189 CONC	0.320 ABS	ABS ABS
3	0.131 ABS 1.317 CONC	0.132 ABS	ABS ABS

Figure 6-5. Plotter Output Obtained by Pressing the Print List, Scaling and Measure keys. The measure key was pressed three times, for the three sample results shown.

-
2. **Auto Set** - The **Auto Set** key can be used to reset the baseline to 0.000 ABS.
 3. **Scaling** - When the **Scaling** key is pressed, the conditions defined in Phases A through E are printed on the printer/plotter. The calibration curve is also drawn, as shown in Figure 6-5. Note: The calibration curve will not be drawn if the (3) FACTOR option was selected.
 4. **Print List** - The **Print List** key can be used to print a header for the analysis, as shown at the top of Figure 6-5. The bandwidth and time constant are also printed.
 5. **Display List** - The **Display List** key is used to display the Time Constant, the status of Auto Set, and the status of Chart Set on the CRT. If **Display List** is pressed again, the sample absorbance and concentration data are returned to the CRT.
 6. **Bandwidth** and **Time Constant** - These parameters can be changed from this page of the Menu.
 7. **Sample Number** - The sample number is displayed on the CRT and printed on the printer/plotter along with each measurement. It is incremented automatically. The **Sample Number** key can be used to set the value of the Sample Number manually, before a measurement.
 8. **Chart Set** - The **Chart Set** key can be set to (2) OFF to inhibit the automatic printing of each measurement on the printer/plotter.

6.7.2 PROCEDURE

1. To create a header for the sample measurements, press **Print List**.
2. Place the sample in the sample holder, and press **Measure**. The results will automatically be displayed on the CRT and printed on the printer/plotter. Note that if the two or three wavelength procedure was selected in Phase A, the absorbance measured at each wavelength is printed on the printer/plotter. However, only the calculated absorbance, Δ ABS is displayed on the CRT.
3. Repeat step 2 until all the samples have been measured.
4. Press **Scaling** to produce a permanent record of the calibration curve data.

-
5. The **Method** key can be used to exit the quantitative analysis program, or to re-enter Method 4 from the beginning of the Menu. The calibration curve data will not be erased until the instrument power is turned off.

7.0 METHOD 5 - TRANSMITTANCE OR ABSORBANCE MEASURED AT FIXED WAVELENGTH INCREMENTS

7.1 DESCRIPTION

In this method, the absorbance or transmittance of a sample is measured at fixed wavelength increments within a specified scanning range. For example, it is possible to scan the region from 500 - 400 nm, collecting one data point every 10 nm. The resulting table of data is printed on the printer/plotter as shown in Figure 7-1.

DATE: __, __, __	OPERATOR: _____	SAMPLE: _____		
CELL: _____	TEMPERATURE: _____	SOLVENT: _____		
	HUMIDITY: _____	CONC: _____		
DATA MODE	:	(1) %T		
BAND WIDTH	:	2.00 nm		
TIME CONST	:	0.4 sec		
λ SET	:	500.0 400.0 nm		
DELTA λ	:	100		
λ SCALE	:	5 nm/cm		
SCAN SPEED	:	100 nm/min		
CYCLE NO	:	1		
	DATA	MODE	λ (nm)	CYCLE NO
	89.9	%T	500.0 nm	1
	83.4	%T	490.0 nm	1
	86.1	%T	480.0 nm	1
	82.2	%T	470.0 nm	1
	22.4	%T	460.0 nm	1
	38.7	%T	450.0 nm	1
	78.1	%T	440.0 nm	1
	87.8	%T	430.0 nm	1
	73.3	%T	420.0 nm	1
	87.6	%T	410.0 nm	1

Figure 7-1. Parameter Listing and Data for an Incremental Scan.

Scan increments as small as 0.01 nm and as large as the full scanning range of the instrument can be selected. Up to 2000 data points can be collected in a single scan.

Two parameters are used to select the scan increment, λ scale and $\Delta\lambda$. Because of the 2000 point per scan limit, and the limits on the data acquisition rate of the instrument, the value chosen for the scan increment will also affect the choice of scan range and scan speed. The relationship between these parameters are shown in Table 7-1.

Table 7-1. Interdependence of Scan Parameters.

λ Set Range (nm)	Scan Speed (nm/min)	Minimum Scan Increment (nm)	λ Scale (nm/cm)
0.5 - 20.0	4 - 10	0.01	0.5
1.0 - 40.0	4 - 20	0.02	1
2.0 - 81.0	4 - 40	0.04	2
5.0 - 204.0	4 - 100	0.10	5
10.0 - 408.0	4 - 200	0.20	10
20.0 - 725.0	4 - 400	0.40	20
40.0 - 725.0	4 - 400	0.80	40

λ scale sets the minimum scan increment for the experiment. These are the same scan increments used during spectral acquisition in Method 1. To obtain larger increments, $\Delta\lambda$ can be used to enter an integer multiplier in the range of 1 to 2000. The scan increment for the experiment is then given by equation 7-1.

$$\text{scan increment} = \Delta\lambda \times \text{minimum scan increment} \quad (7-1)$$

This process of parameter selection can be illustrated using the data in Figure 7-1. The selected scan range is 100 nm (500nm - 400nm). A scan speed of 100 nm/min is used. Table 7-1 shows that a λ scale value of 5 nm/cm is compatible with this range and scan speed. This results in a minimum scan increment of 0.1 nm. To obtain one data point every 10nm, a multiplier of 100 is selected for $\Delta\lambda$.

It should be noted that several combinations of λ scale and $\Delta\lambda$ can be used to obtain the same scan increment. Under the constraints of scan range and scan speed cited in the example above, any of the following combinations will yield a scan increment of 10 nm:

λ scale		5		10		20
$\Delta\lambda$		100		50		25

Note: The combination λ scale = 40, $\Delta\lambda = 12.5$ can not be used, since $\Delta\lambda$ must be an integer.

A detailed operating procedure for Method 5 is given in the next section.

7.2 PROCEDURE

1. Press the **Method** , **5** and **Enter** keys. A listing of the parameters used in this method will appear on the CRT as shown in Figure 7-2.

METHOD 5: INCREMENTAL WAVELENGTH MODE	
DATA: 0.000 ABS λ :500.0 nm BANDWIDTH:2.00 nm	
<hr/>	
Time Const	: 0.4 sec
Auto Set	: (1) ON
λ Set	: 900.0 ~ 200.0 nm
$\Delta\lambda$: 1
λ Scale	: 20 nm/cm
Scan Speed	: 100 nm/min
Cycle No.	: 1
Cycle Time	: 0 min 0 sec
<hr/>	

Figure 7-2. Method 5 Operating Parameter Display

2. To select the upper and lower wavelength limits for the scan, use the **λ Set** key. The difference between the upper and lower wavelength limits is the λ Set range. This range determines the maximum scan speed and the minimum wavelength increment between data points, as shown in Table 7-1.
3. Using Table 7-1, select values for **Scan Speed** and **λ Scale** that are consistent with the the λ Set Range. Note that there is considerable overlap of the λ Set Ranges. This provides for greater flexibility in

parameter selection. When more than one set of conditions can be used for a given λ Set Range, the following guidelines can aid in the parameter selection:

- a. To maximize Scan Speed, select the largest λ Scale value allowable.
 - b. To minimize the wavelength increment between successive data points, select the smallest λ Scale value allowable.
4. The $\Delta\lambda$ key is used to obtain a scan increment larger than the minimum values listed in Table 7-1. $\Delta\lambda$ can be an integer in the range of 1 to 2000. This integer is then multiplied by the minimum scan increment. To calculate the $\Delta\lambda$ value needed to obtain a desired increment, use equation (7-2).

$$\Delta\lambda = \frac{\text{desired scan increment}}{\text{minimum scan increment}} \quad (7-2)$$

The calculated $\Delta\lambda$ value must be rounded off to the nearest integer. Table 7-2 lists $\Delta\lambda$ values for a few selected Scan Increments. To use the table, locate the desired increment, then read across the table to find the $\Delta\lambda$ value appropriate for the λ Scale Value selected previously.

5. The Cycle Number and Cycle Time keys can be used to program a series of replicate measurements. The cycle time is measured from the beginning of one cycle to the beginning of the next. Both minutes and seconds can be entered. For example, to select a cycle time of 2 minutes, 5 seconds, enter "002-05". Two digits must always be entered for the seconds.
6. When all of the parameters have been selected, the Print List key can be used to obtain a header for the sample, and a permanent record of the parameters used, as shown in Figure 7-1.
7. Place the sample in the sample compartment and press Measure . The scan will begin and the data will be printed out, as shown in Figure 7-1.
8. Note: Using this method, up to 2000 data points can be collected. A maximum data collection rate of 1000 points per minute can be achieved. This data cannot be printed on the printer/plotter in real time, since a printing time of approximately 2 seconds per data point is required. When the scan is completed, data will continue to be printed until all the data points have been recorded. If the Stop key is used to abort a scan, the printer will continue to list all data collected up to the time the Stop key was pressed.

Table 7-2. $\Delta\lambda$ Values for Selected Scan Increments

Desired Scan increment (nm)	λ Scale: 0.5 (nm/cm)	1	2	5	10	20	40
0.1	10	5	-	1	-	-	-
0.2	20	10	5	2	1	-	-
0.4	40	20	10	4	2	1	-
0.6	60	30	15	6	3	-	-
0.8	80	40	20	8	4	2	1
1.0	100	50	25	10	5	-	-
2.0	200	100	50	20	10	5	-
4.0	400	200	100	40	20	10	5
6.0	600	300	150	60	30	15	-
8.0	800	400	200	80	40	20	10
10.0	1000	500	250	100	50	25	-
20.0	2000	1000	500	200	100	50	25
40.0	-	2000	1000	400	200	100	50



8.0 METHOD 6 - WAVELENGTH PROGRAMMING

8.1 DESCRIPTION

In this method, the transmittance or absorbance of a sample can be measured at up to ten programmable wavelengths. A point plot of the data is drawn on the printer/plotter. Below each plotted point, the measured photometric value is printed out.

8.2 PROCEDURE

1. Press the **Method** , **6** and **Enter** keys. The CRT will display the parameters used in this method, as shown in Figure 8-1.
2. The **λ Set** key is used to select the wavelengths used in the experiment. Up to ten wavelengths can be entered. As each wavelength is entered, it will appear in the appropriate space in the parameter list. When the last desired wavelength is entered, press the **+** and **Enter** keys to complete the parameter entry procedure. If fewer than ten wavelengths are entered, the unused values are automatically cleared.

```
METHOD 6: WAVELENGTH PROGRAMMING

DATA: 57.1%T  λ: 418.7 nm      Bandwidth: 2.00 nm

-----

TIME CONST   : 0.4 sec
λ SET        λ0: 637.5, 536.2, 484.5, 460.0, 453.0
              λ5: 418.7, 385.9, 360.9, 333.7, 287.5
%T ABS SCALE : 0.0-100.0%T
SCALING      : (1) ON
CYCLE NO.    : 2
CYCLE TIME   : 0 min 0 sec

-----
```

Figure 8-1. Operating Parameters for Method 6, Wavelength Programming.

3. When all of the parameters have been set, the **Print List** key can be used to obtain a header for the experiment, and a listing of the parameters used. A sample output is shown in Figure 8-2.
4. Place the sample in the sample chamber and press **Measure**. If the Scaling status is (1) ON, then a photometric axis will be automatically drawn on the printer/plotter. After the data has been collected, it is plotted as shown in Figure 8-2. A 0.5 cm line is drawn to indicate the photometric value, and a small circle is drawn at the end of the line. At the edge of the graph, directly opposite the plotted circle, the photometric value is printed out.
5. The **Cycle Number** and **Cycle Time** keys are used to perform replicate experiments automatically. The cycle time is measured from the beginning of one cycle to the beginning of the next. The time is entered in minutes and seconds, separated by a hyphen. For example, to select a time of 2 minutes 5 seconds, enter "002-05". Two digits must always be entered for the seconds.

DATE: __, __, __ OPERATOR: _____ SAMPLE: _____
CELL: _____ TEMPERATURE: _____ SOLVENT: _____
HUMIDITY: _____ CONC: _____

DATA MODE : (2) ABS
BAND WIDTH : 0.50 nm
TIME CONST : 0.4 sec
 λ SET : 637.5, 536.2, 484.5, 460.0, 453.2
 : 418.7, 385.9, 360.9
%T ABS SCALE : 0.000 ~ 1.000 ABS
CYCLE NO : 1

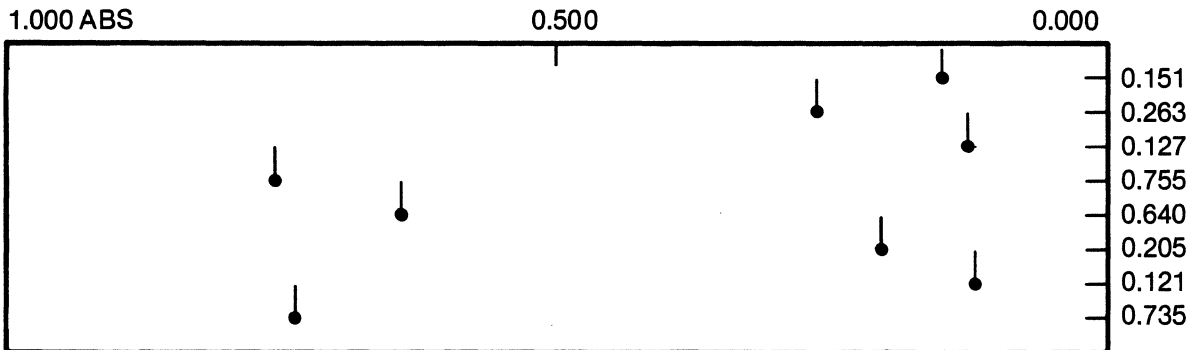


Figure 8-2. Plotter Output for Method 6, Cycle Number = 1. The photometric value for each measurement is printed along the right edge of the chart.

-
6. When replicate cycles are performed, the photometric axis is drawn only at the beginning of the first cycle. The data points collected at each wavelength are connected by a line segment at the end of each cycle. This allows the change in absorbance or transmittance at each wavelength to be followed easily from cycle to cycle. An example of a two-wavelength experiment, repeated for 10 cycles, is shown in Figure 8-3.
 7. Note: Although the 10 wavelengths used in this method can be entered in any order, it is most efficient to enter the values in decreasing order. This helps to minimize the overall scanning time. For example, if four wavelengths are selected in the order 500.0, 900.0, 200.0 and 850.0 nm, the total distance scanned is 2100 nm per cycle, since the instrument always returns to the initial wavelength at the end of the scan. If instead the wavelengths were entered as 900.0, 850.0, 500.0 and 200.0, the total scanning distance is only 1400 nm.

DATE: __, __, __ OPERATOR: _____ SAMPLE: _____
CELL: _____ TEMPERATURE: _____ SOLVENT: _____
HUMIDITY: _____ CONC: _____

DATA MODE : (1) %T
BAND WIDTH : 0.50 nm
TIME CONST : 0.4 sec
 λ SET : 352.0 , 319.0
:
%T ABS SCALE : 0.0 ~ 50.0 %T
CYCLE NO : 10

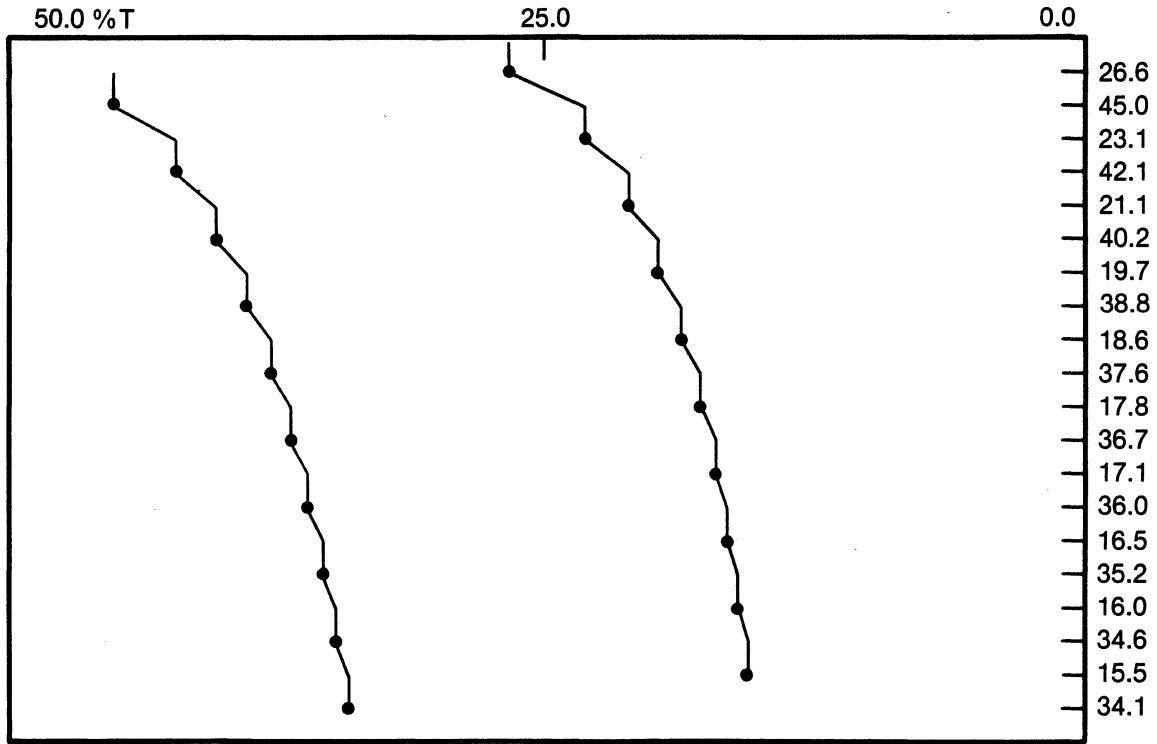


Figure 8-3. Plotter Output for Method 6, Cycle Number = 10. The photometric value for each measurement is printed along the right edge of the chart.



9.0 METHOD 11 - DATA PROCESSING OF SPECTRA

9.1 INTRODUCTION

A spectrum collected using Method 1, can be stored in one of the five memory locations available in the 9420/30. Method 11 is used to perform the following functions on the stored spectrum:

1. Display data on the CRT.
2. Plot data.
3. Transfer or exchange spectra between memory locations.
4. Perform arithmetic operations.
5. Smooth data.
6. Differentiate data.
7. Locate peaks in the spectrum.

The sections that follow describe each of these functions in detail.

Note: Method 11 cannot be used to collect spectra. It is used only to process the spectra collected using Method 1.

9.2 STORING SPECTRA IN MEMORY

9.2.1 DESCRIPTION

There are five memory locations available for storing spectra in the 9420/30. Each memory location can hold one spectrum, containing up to 2048 data points. These are volatile memories. Spectra will remain stored in memory as long as the instrument power is on.

The five memories are numbered 0 through 4. When a spectrum is collected using Method 1, the data is automatically stored in memory 1. Each time a new spectrum is collected, the previous contents of memory 1 are erased. Therefore, to save a spectrum, it is necessary to move the data out of memory 1 before the next spectrum is collected.

Two command keys are used to move spectra between memories, **TRANSFER** and **EXCH**.

The **TRANSFER** key moves data from one memory (the source) into a second memory (the destination). The previous contents of the destination memory are erased. The source memory is not erased by this command, therefore two copies of the transferred spectrum will be present in the system after this command is executed.

The **EXCH** key exchanges the contents of two memories. Neither spectrum is erased by this command.

Note: Memory 0 is used by the 9420/30 during Method 55 - Spectral Ratio/Subtract Mode. If Method 55 is in use, then Memory 0 should not be used for spectral storage in Method 11.

9.2.2 PROCEDURE

1. To Transfer data from one memory to another, press the following key sequence:

Memory **N1** **Transfer** **Memory** **N2** **Enter**

N1 is the number of the source memory. If the data was just collected using Method 1, N1 = 1.

N2 is the number of the destination memory. Remember that the previous contents of N2 will be erased.

Both N1 and N2 must be integers (0-4).

-
2. To exchange the contents of two memory locations, press the following key sequence:



Again, Both N1 and N2 must be integers (0-4).

9.3 DISPLAYING STORED SPECTRA ON THE CRT

9.3.1 DESCRIPTION

The stored spectra can be displayed on the CRT as shown in Figure 9-1. A cursor appears on the CRT, and can be positioned at any point on the spectrum. The wavelength at which the cursor is positioned is displayed in the upper left corner of the CRT, together with the transmittance or absorbance measured at that wavelength.

The scale used to display the data can be changed, allowing the spectrum to be expanded or compressed along the photometric axis. This feature is used to bring large spectral features back on scale, or to expand smaller features that might be hidden in the baseline.

Each spectrum stored in memory has an associated list of parameters. These parameters can be displayed on the CRT, as shown in Figure 9-2.

The following items are demonstrated in the procedure that follows:

1. Display of spectral data.
2. Cursor Positioning.
3. Scale expansion.
4. Display of parameter lists.

Note: Only one spectrum can be displayed on the CRT at a given time. Overlay plots of the data can be obtained with the printer/plotter only.

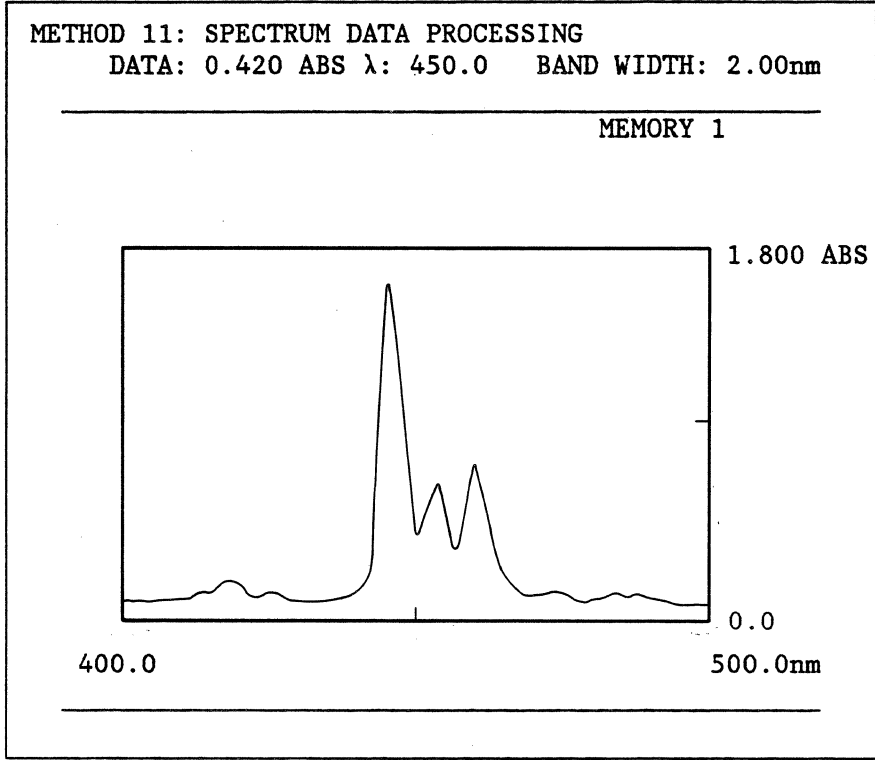


Figure 9-1. CRT Display of a Stored Spectrum

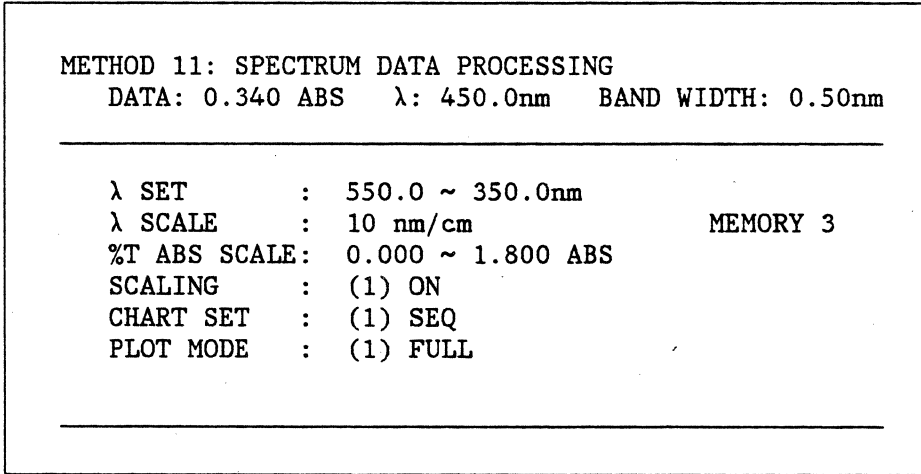


Figure 9-2. CRT Display of a Parameter List

9.3.2 PROCEDURE

1. To display a spectrum on the CRT, press the following keys:

Memory

N

Enter

N is the number of the memory in which the data is stored. N must be an integer (0-4). The data will appear on the CRT as shown in Figure 9-1.

2. Initially, the cursor will be positioned in the center of the spectrum. To move the cursor, press either of the following keys:

Cursor →

 or

Cursor ←

The cursor can be moved one point at a time by pressing the key momentarily, then releasing it. To move the cursor continuously, simply hold the key down. After the first five points, the cursor will begin to skip to every fifth point, allowing faster movement from one part of the spectrum to another.

3. The position of the cursor is continuously displayed in the upper left corner of the CRT. To print this value on the printer/plotter, press the Print key.
4. To change the scale of the spectrum, the following procedure is used:
 - a. Press the %T/ABS Scale key, and select the desired scale. When the new scale is entered, the CRT will be erased, and a new set of axes will be drawn.
 - b. To re-display the spectrum with the new scale, enter the command:

Memory

N

Enter

as in Step 1 above.

5. To display the parameter list associated with any spectrum, the following procedure is used:
 - a. Display the spectrum as shown in Step 1 above.

-
- b. Press the **Display List** key. The spectrum will be erased, and the parameters associated with it will be displayed, as shown in Figure 9-2.
 - c. To return to the spectral display, press the **Display List** key again.

Note: The bandwidth displayed in the upper right corner of the CRT is not stored with the data. The value displayed reflects the last bandwidth used before entering Method 11, not the value used when the data was collected.

9.4 PLOTTING STORED SPECTRA

9.4.1 DESCRIPTION

The stored spectra can be plotted on the printer/plotter as shown in Figure 9-3. The following functions are demonstrated in the procedure that follows:

1. Plotting the spectrum.
2. Scale expansion/compression.
3. Overlay plotting.
4. Printing the parameter list.

DATE: __, __, __ OPERATOR: _____ SAMPLE: _____
CELL: _____ TEMPERATURE: _____ SOLVENT: _____
HUMIDITY: _____ CONC: _____

DATA MODE : (1) %T
λ SET : 550.0 ~ 350.0 nm
λ SCALE : 20 nm/cm
%T ABS SCALE : 0.0 ~ 90.0 %T

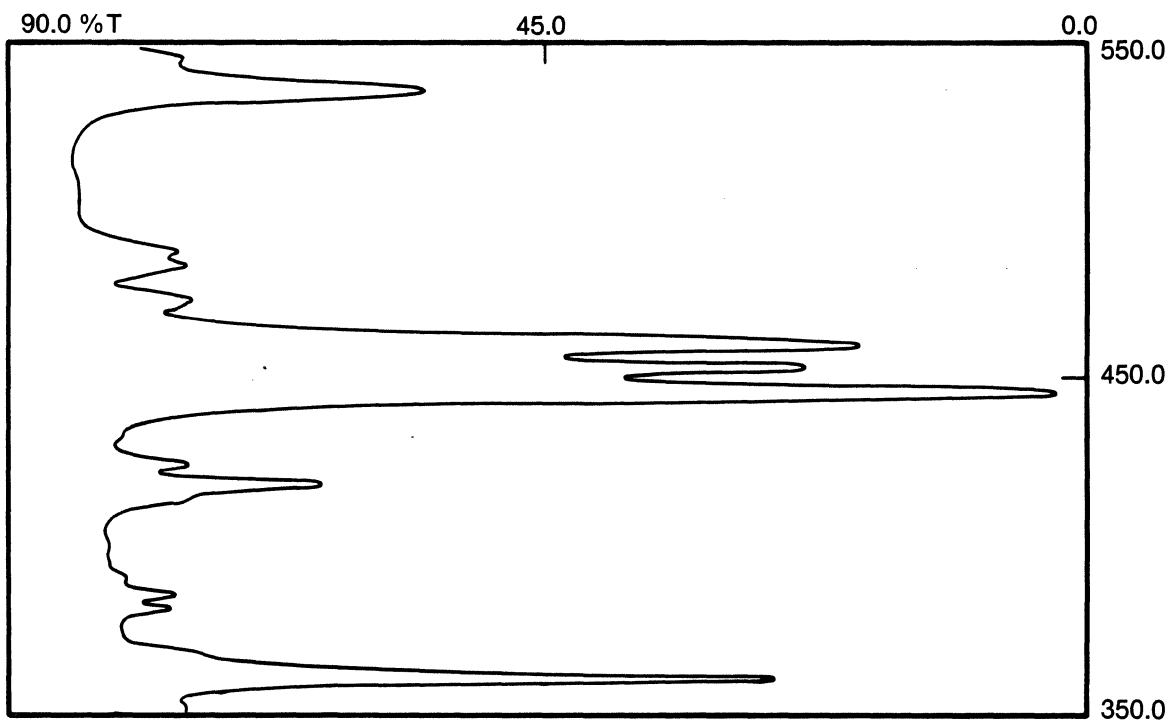


Figure 9-3. Plot of Stored Spectrum With Parameter List

9.4.2 PROCEDURE

1. Display the spectrum on the CRT, as described in section 9.3. This will aid in the selection of the appropriate scale, and will specify the memory to be used for listing parameters.
2. To select a new scale, press the **%T/ABS Scale** key, and enter the new value. This allows expansion or compression of the data along the photometric axis.
3. Press the **Chart Set** key. Three choices are displayed - (1) SEQ, (2) OVERLAY, and (3) OFF. Selections are made as follows:
 - a. If spectra will be plotted sequentially with only one spectrum drawn on each set of axes, select (1) SEQ.
 - b. If spectra will be overlaid on a single set of axes, as shown in Figure 9-4, select (2) OVERLAY.
4. Press the **Plot Mode** key to select the line type for the plot. The choices are - (1) FULL, (2) DOTTED, (3) BROKEN, and (4) CHAIN.
5. To print a header for the data and a listing of the parameters associated with the spectrum, press the **Print List** key. A sample of the print out obtained is shown in Figure 9-3.
6. Press the **Scaling** key. Three choices are displayed - (1) ON, (2) OFF, and (3) DRAW. Selections are made as follows:
 - a. If sequential plots are being made, select (1) ON to automatically draw the axis each time a plot is made.
 - b. If overlay plots are being made, select (3) DRAW. Two things will happen. First, the 9420/30 will draw the axes on the printer/plotter and rewind the chart paper to the beginning of the axes. Secondly, the Scaling status will be set to (2) OFF, automatically. This prevents the axes from being redrawn each time a plot is made.

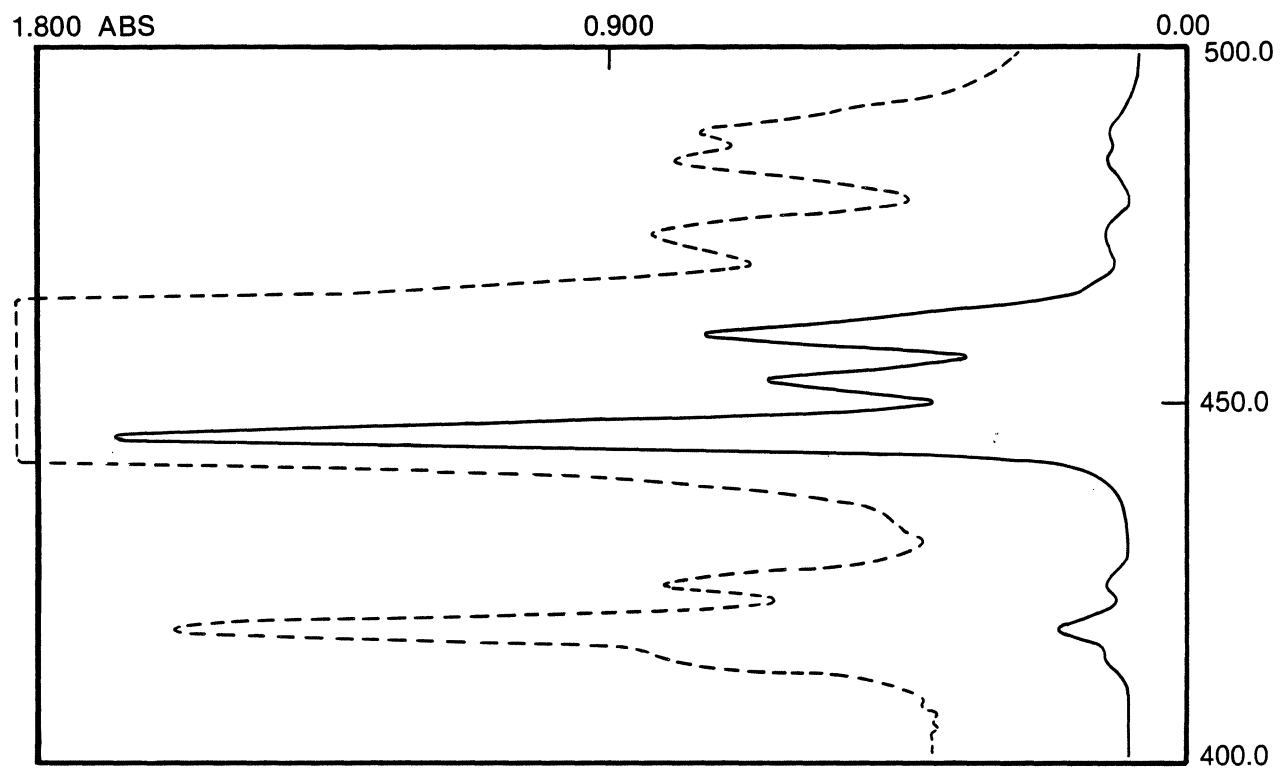
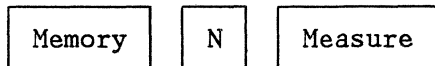


Figure 9-4. Overlay Plotting of Spectra

7. To plot the data, press the following keys:



N is the number of the memory in which the data is stored. N must be an integer (0-4).

8. If spectra are being overlaid, repeat Step 7 until all the spectra are plotted. If desired, the **Plot Mode** key can be used to change the line type between plots.

9.5 ARITHMETIC OPERATIONS

9.5.1 DESCRIPTION

The arithmetic operations of addition, subtraction, multiplication and division can be performed on stored spectra. Constants entered from the keypad can be used in these operations. Two spectra collected under identical conditions can also be combined arithmetically.

The following limitations apply to the arithmetic operations:

1. Values larger than $\pm 655.30\%T$ or ± 6.5530 ABS cannot be calculated. If an operation results in a value outside these limits, the result is set equal to the upper or lower limit (e.g., $10 \times 70.0\%T$ would return a value of $655.3\%T$).
2. Decimal values are truncated beyond ± 0.0001 ABS and $\pm 0.01\%T$.
3. If two spectra are not collected under identical conditions, they cannot be combined arithmetically. The following error codes are used as a warning if the spectra are not compatible:
 - E010 - The two spectra were not collected in the same photometric mode, $\%T$ or ABS.
 - E011 - The spectra were not collected with the same λ Scale value. Therefore, the spacing between data points on the wavelength axis is different for the two data sets.

E012 - The spectra were not collected with the same λ Set value. Therefore the data sets do not cover the same wavelength region.

9.5.2 PROCEDURE

1. The following procedure is used to perform arithmetic operations with a constant:
 - a. Select the spectrum to be used in the calculation by pressing the following keys:

Memory

N1

Where N1 is the memory location (0-4) that contains the data. (Do not press Enter yet.)

- b. Select the operation to be used by pressing one of the following keys:

x

 ,

÷

 ,

+

 or

-

- c. Enter the constant to be used from the numeric key pad. Use the - key to indicate a negative constant. (If a negative value less than 1 is entered, a leading zero is needed, e.g., -0.01.)
 - d. Select the memory in which the result is to be stored by pressing the following keys:

Transfer

Memory

N2

N2 indicates the memory location (0-4) for storage. If N1 = N2 the original data will be replaced with the new data.

- e. Press

Enter

 to complete the command sequence.

Example: To multiply the spectrum in memory 1 by 5, and store the result in memory 2, the following command is used:

Memory 1 x 5 Transfer Memory 2 Enter

2. The following procedure is used to perform arithmetic operations with two spectra:

a. Enter the location of the first spectrum by using the following keys -

Memory N1

b. Select the operation by pressing one of the following keys -

x , ÷ , + or -

c. Enter the location of the second spectrum by using the following keys -

Memory N2

d. Transfer the result to another memory location by using the following key sequence -

Transfer Memory N3

e. Press Enter to complete the command sequence.

Example: To subtract the spectrum in memory 1 from the spectrum in memory 2 and store the result in memory 3, the following command is used:

Memory 2 - Memory 1 Transfer Memory 3 Enter

9.6 SPECTRAL SMOOTHING

9.6.1 DESCRIPTION

Smoothing routines are often used to improve the signal-to-noise ratio of spectral data. These routines serve to filter out the higher frequency "noise", while minimizing any distortion of the underlying "signal".

The 9420/30 uses a seven point weighted average smoothing algorithm. A seven point segment of the spectrum is used to calculate a new, smoothed value for the data point in the center of the seven point window. The weighted average is equivalent to performing a least-squares fit of the data to a third-order polynomial, and calculating a new center point.

After each new point is calculated, the window is shifted over one point, and the process is repeated. This continues until the entire spectrum is smoothed. Figure 9-5 gives a pictorial representation of this process.

The degree of smoothing obtained is a function of the width of the smoothing window. A wider window gives a greater level of noise reduction. Since the 9420/30 has a fixed window size of seven points, wider effective windows are obtained by skipping data points, as shown in Figure 9-6. M is an integer that is specified when the smoothing routine is used. The effect of M on an actual data set is shown in Figure 9-7. The signal-to-noise ratio of the spectrum noticeably improves as M is increased from 3 to 5.

While larger values of M result in greater noise reduction, it should be noted that distortions of the spectrum can also occur at larger M values. In general, the smallest M which gives adequate noise reduction should be used.

When selecting an M value, it is useful to consider the actual width of the smoothing window in nanometers. This value depends on both M and on the wavelength increment between successive data points in the scan. Table 9-1 shows the effective smoothing width, in nanometers, for M=1 to 5, as a function of λ Scale.

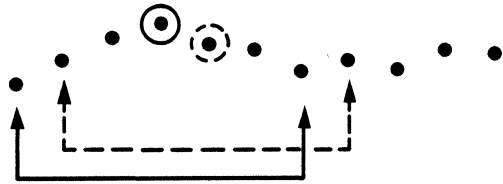


Figure 9-5. Seven-point Smoothing

Note: This is a pictorial representation of a seven-point, sliding, smoothing window. The first seven points are used to calculate a new center point (solid lines). The window is then shifted one point to the right, and the process is repeated (dashed lines).

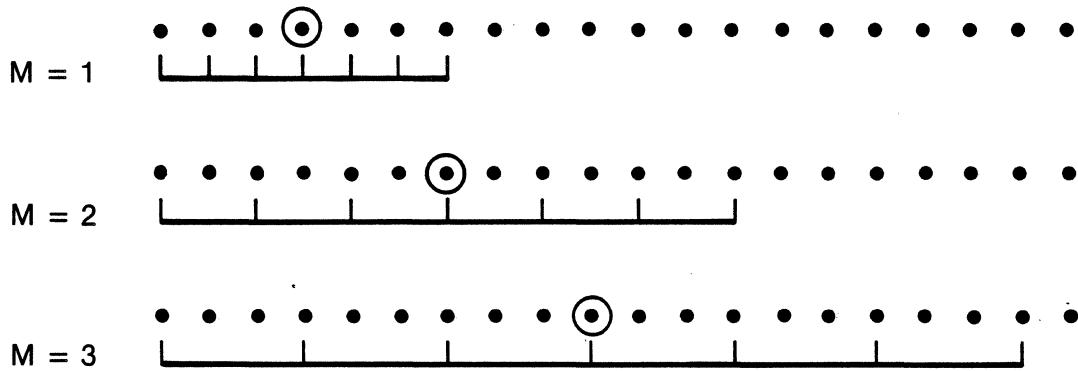


Figure 9-6. Effect of M on the Seven-point Smoothing Window

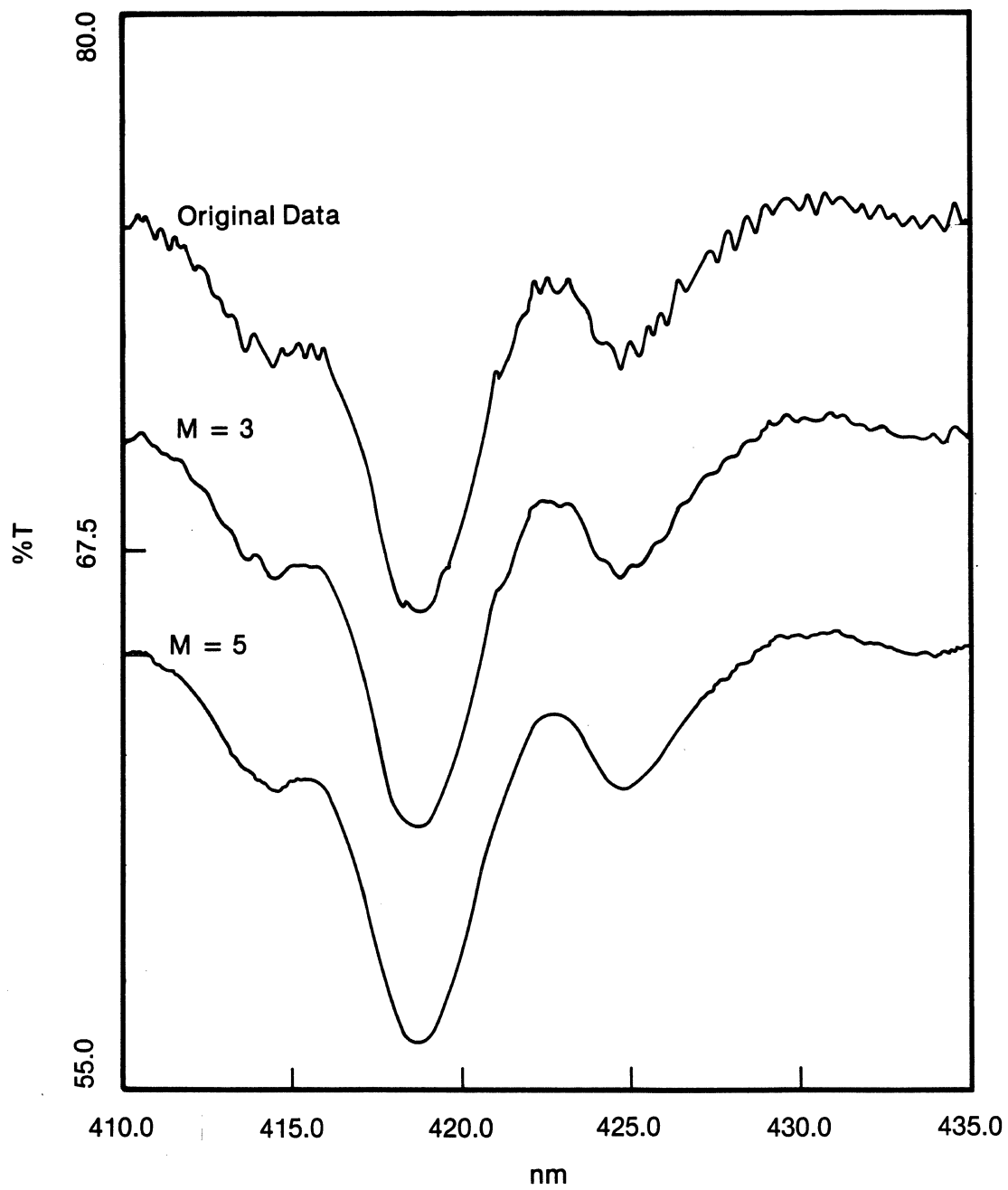


Figure 9-7. Effect of M on the Smoothing Function

Table 9-1. Effective Smoothing Width (nm)

M	λ Scale (nm/cm)						
	0.5	1.0	2.0	5.0	10	20	40
1	0.06	0.12	0.24	0.60	1.20	2.40	4.80
2	0.12	0.24	0.48	1.20	2.40	4.80	9.60
3	0.18	0.36	0.72	1.80	3.60	7.20	14.40
4	0.24	0.48	0.96	2.40	4.80	9.60	19.20
5	0.30	0.60	1.20	3.00	6.00	12.00	24.00

To Calculate smoothing widths that are not given in the table, use equation (9-1).

$$\text{Effective Smoothing Width} = 6M \times \frac{\lambda\text{Scale}}{50} \quad (9-1)$$

As a "rule-of-thumb", the smoothing width should not exceed 2 times the full width at half maximum (FWHM) of the narrowest spectral feature of interest. Using this criterion, it is clear that larger values of M can be used when the data density is high (small values of λ Scale).

The data shown in Figure 9-7 was collected using a λ Scale value of 2 nm/cm. This gives a smoothing width of 1.2 nm, when M=5. This is well within the criterion stated above.

Note: The λ Scale value is fixed when the data is collected, and cannot be changed in Method 11.

9.6.2 PROCEDURE

1. Display the spectrum to be smoothed on the CRT, as described in Section 9.3.
2. Using the criteria given in the discussion above, estimate the value needed for M.

-
3. To smooth the spectrum, enter the following key sequence:

Memory N1 Smooth M Transfer Memory N2 Enter

N1 and N2 are integers (0-4). Memory N1 contains the data to be smoothed. Memory N2 is used to store the smoothed data. M must be an integer (1-99).

4. Display the smoothed spectrum on the CRT using the following key sequence:

Memory N2 Enter

5. If greater smoothing is required, increase M and repeat step 3. If distortion of the spectrum occurs, decrease M, and repeat step 3.

9.7 DIFFERENTIATION OF SPECTRA

9.7.1 DESCRIPTION

Differentiation is frequently used to aid in resolving poorly defined spectral features. Although the derivative spectrum does not contain any more information than the original data, the derivative is often easier to interpret.

An example of differentiation is shown in Figure 9-8. The solid line shows the transmittance spectrum of holmium oxide glass, from 435 nm to 410 nm. This spectrum was obtained at low resolution (Bandwidth = 5 nm). The small peak, which usually occurs on the high wavelength side of the main band at 418.7 nm, appears here as a shoulder on the main band.

The dotted line shows the 4th derivative of the transmittance spectrum. The derivative shows a sharp peak for both the main band and the shoulder. The derivative can be used to easily locate the wavelength maximum for each feature.

The 9420/9430 software can be used to calculate the first through the fourth order derivatives for any stored spectrum. The derivatives are calculated using a sliding, least-squares fit similar to that used by the smoothing routine (see Figure 9-5, Section 9.6). The width of the sliding window can be varied by

DATA MODE : (1) %T
BAND WIDTH : 5.00 nm
TIME CONST : 0.4 sec
 λ SET : 435.0 ~ 410.0 nm
 λ SCALE : 2 nm/cm
SCAN SPEED : 40 nm/min
%T ABS SCALE : 55.0 ~ 80.0 %T
CYCLE NO. : 1

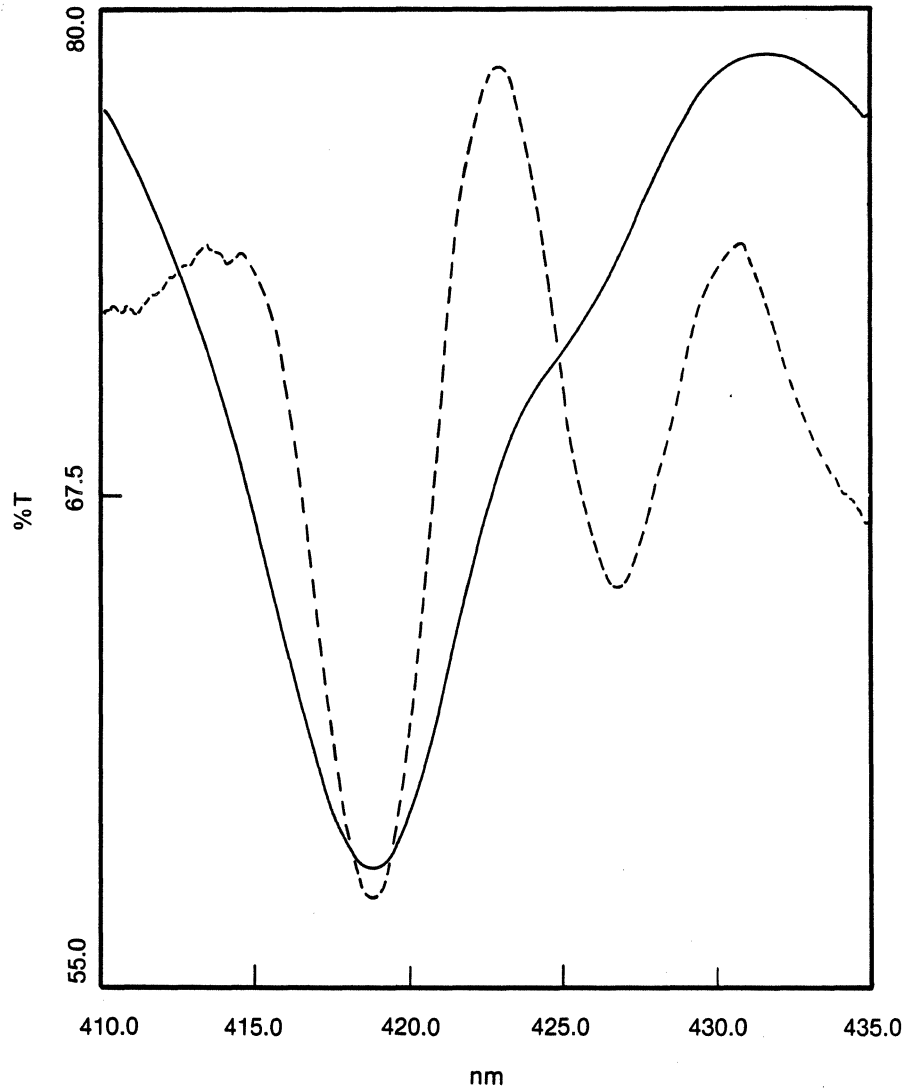


Figure 9-8. Example of Spectrum Differentiation

NOTE: Solid Line - Transmittance of Holmium Oxide Glass
Broken Line- Fourth Derivative Spectrum, M=50 (Scaled Data)

specifying the value of the integer M.

NOTE: The M value is not used in the same way by the smoothing and differentiation routines.

The selection of M affects both the signal-to-noise ratio of the resulting derivative, and the intensity of the derivative. Larger M values result in better signal-to-noise ratios and higher intensity derivative spectra. However, if M is too large, distortion of the spectrum will occur. In many cases, M can be selected empirically, increasing or decreasing the value until the desired results are obtained. The derivative spectrum can be displayed on the CRT to determine if the signal-to-noise ratio and intensity are adequate, prior to plotting a hard copy on the thermal printer/plotter.

The optimum value of M depends on two factors: (1) the data density of the original spectrum (defined by the λ Scale value used to collect the data); and (2) the order of the derivative selected (first through fourth). The effect of these factors on the selection of M is illustrated in the following examples:

1. Figure 9-9(a) shows an absorbance spectrum of holmium oxide glass, from 550 nm to 350 nm. The data was collected using a λ Scale value of 10, giving a data density of one point every 0.2 nm. (See Table 9-2.)

Table 9-2. Scan Increment as a Function of the λ Scale Value

λ Scale (nm/cm)	Scan Increment (nm/point)
0.5	.01
1	.02
2	.04
5	.1
10	.2
20	.4
40	.8

Figure 9-9(b) shows the first derivative of this spectrum, calculated with an M value of 2. The effective window size, in nanometers, is a function of both M and the Scan Increment, as shown in equation (9-2).

$$\text{Window Size} = 2M \times \text{Scan Increment} \quad (9-2)$$

In this example, the effective window size is 0.8 nm. This is small enough to prevent distortion of the spectrum, while still providing a good signal-to-noise ratio in the derivative spectrum.

DATA MODE : (2) ABS
BAND WIDTH : 2.00 nm
TIME CONST : 0.4 sec
 λ SET : 550.0 ~ 350.0
 λ SCALE : 10 nm/cm
SCAN SPEED : 1000 nm/min
%T ABS SCALE : 0.000 ~ 1.000 ABS
CYCLE NO. : 1

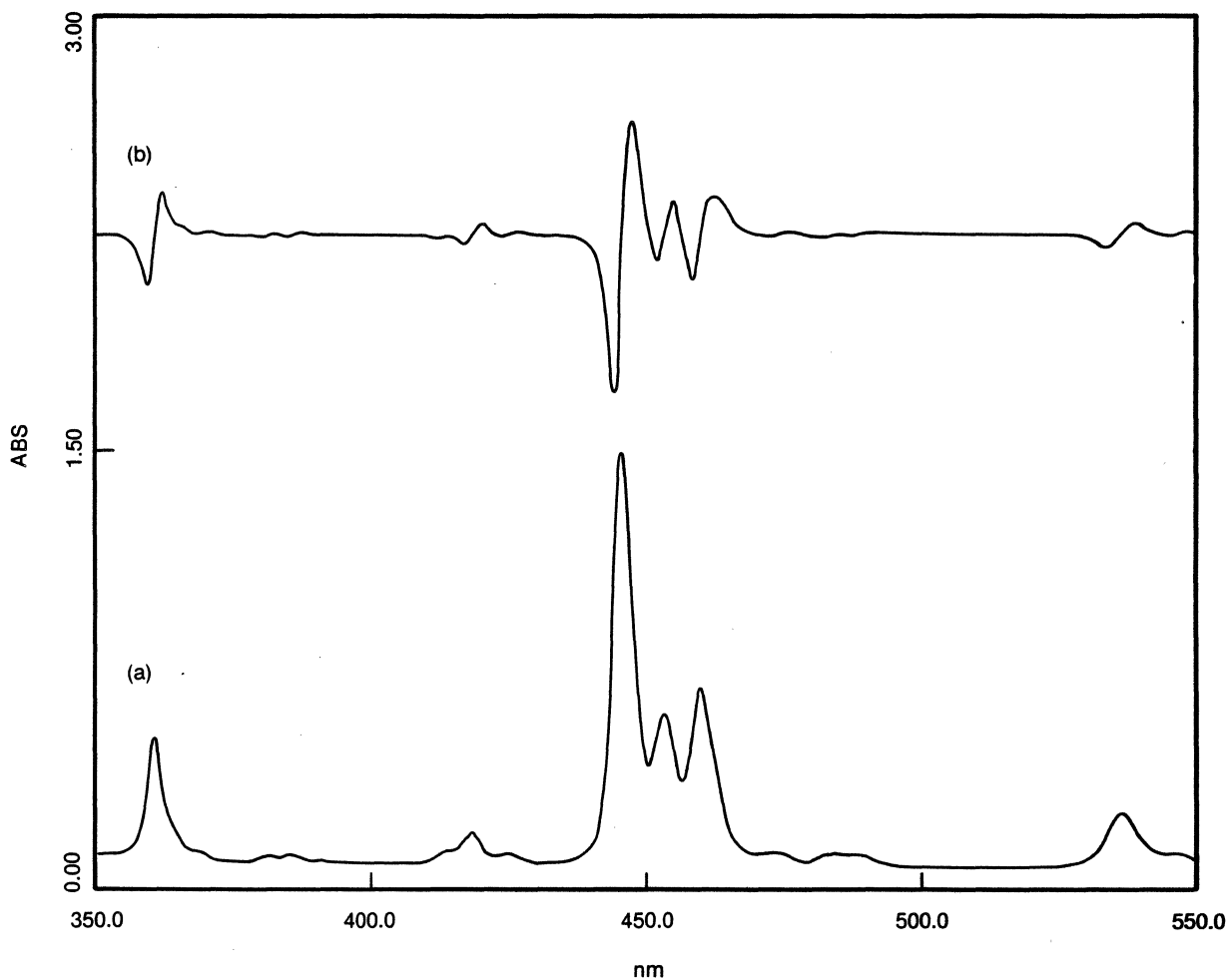


Figure 9-9. First Derivative of Holmium Oxide Spectrum

NOTE: (a) Absorbance of Holmium Oxide Glass.
(b) First Derivative of (a), M=2.

-
2. Figure 9-10(a) shows the absorbance spectrum of holmium oxide glass. Data was collected from 429 nm to 409 nm, using a λ Scale value of 1. The scan increment is one point every 0.02 nm, which is ten times greater than the density used in Figure 9-9.

To obtain the same effective window size as in Figure 9-9, an M value ten times larger was needed. Figure 9-10(b) shows the first derivative obtained with M=20, for an effective window size of 0.8 nm. The signal-to-noise ratio is comparable to that shown in Figure 9-9(b). Note that the derivative data was multiplied by 10 before plotting.

Figure 9-10(c) illustrates the effect of using an M value that is too small. An M value of 2 was used, giving an effective window size of 0.08 nm. Clearly, the signal-to-noise ratio is much worse with the smaller window. In addition, the intensity of the derivative was decreased. Note that a scaling factor of 75 was needed for the derivative data.

3. The optimum value of M also varies with the order of the derivative being calculated. The fourth derivative of the data shown in Figure 9-10(a) was calculated using an M value of 20. The result is shown in Figure 9-11(a). While M=20 gave an excellent signal-to-noise ratio for the first derivative, a larger value is needed for the fourth derivative. Figure 9-11(b) shows the result obtained for M=50. Note that there is a difference of a factor of 10 in the scaling factors used for the plot.

The observations made in the previous examples can be summarized as follows:

1. As the λ scale decreases, the value selected for M must increase to obtain the same signal-to-noise ratio.
2. Higher order derivatives tend to result in higher noise levels. Therefore, larger values of M must be used to compensate for the noise levels.

9.7.2 PROCEDURE

1. Display the spectrum whose derivative is to be calculated on the CRT, as described in Section 9.3.
2. Determine the order (K) of the derivative to be calculated (first to fourth).
3. Using the criteria given in the description above (Section 9.7.1), select the width of the sliding window, M.

BAND WIDTH: 2 nm

λ SCALE : 1 nm/cm

NOTE: (a) Absorbance of holmium oxide glass.

(b) First Derivative of (a) Multiplied by Factor of 10, $M=20$.

(c) First Derivative of (a) Multiplied by a Factor of 75, $M=2$.

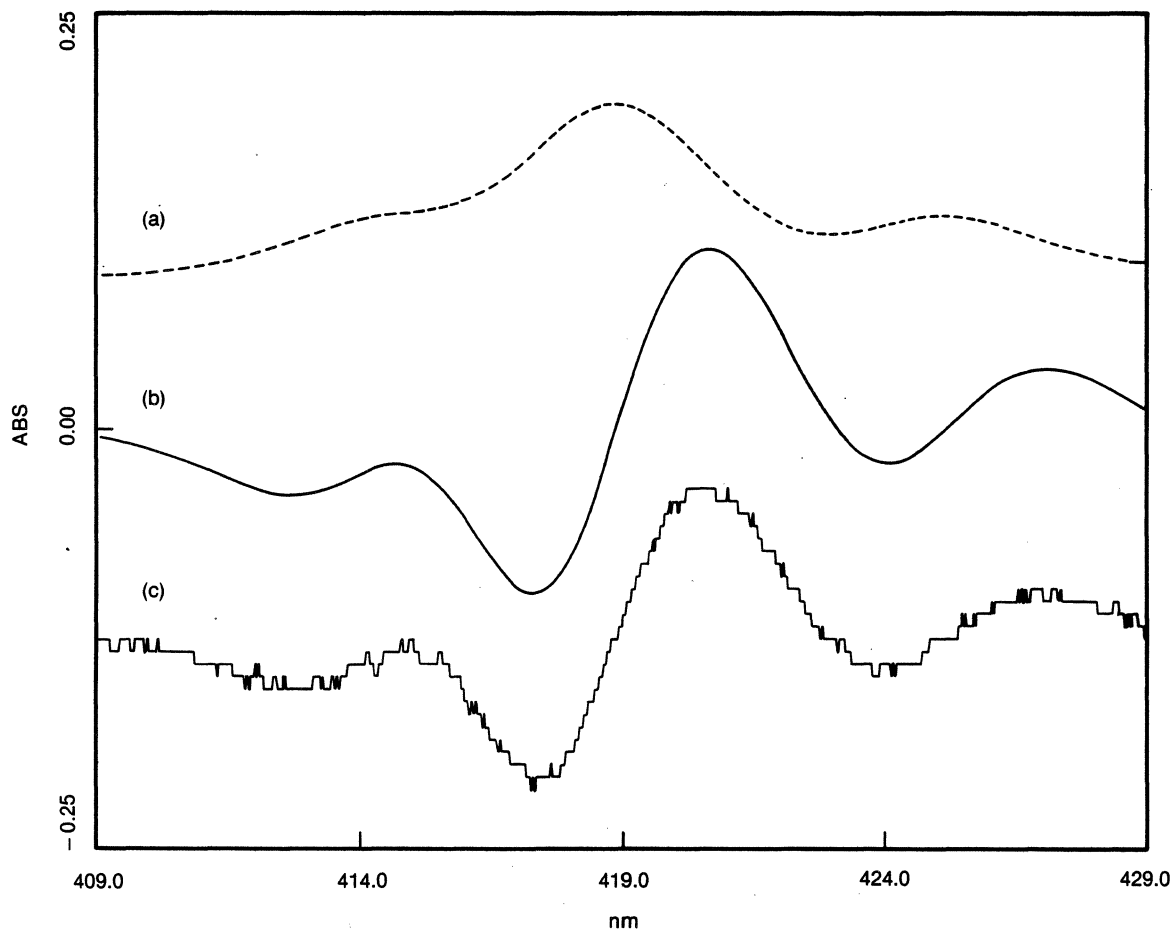


Figure 9-10. Effect of M Value on Differentiation

NOTE: (a) Fourth Derivative of the Spectrum Shown in Figure 9-10(a); multiplied by a factor of 100, M=20.
(b) Fourth Derivative of the Same Spectrum; Multiplied by a factor of 10, M=50.

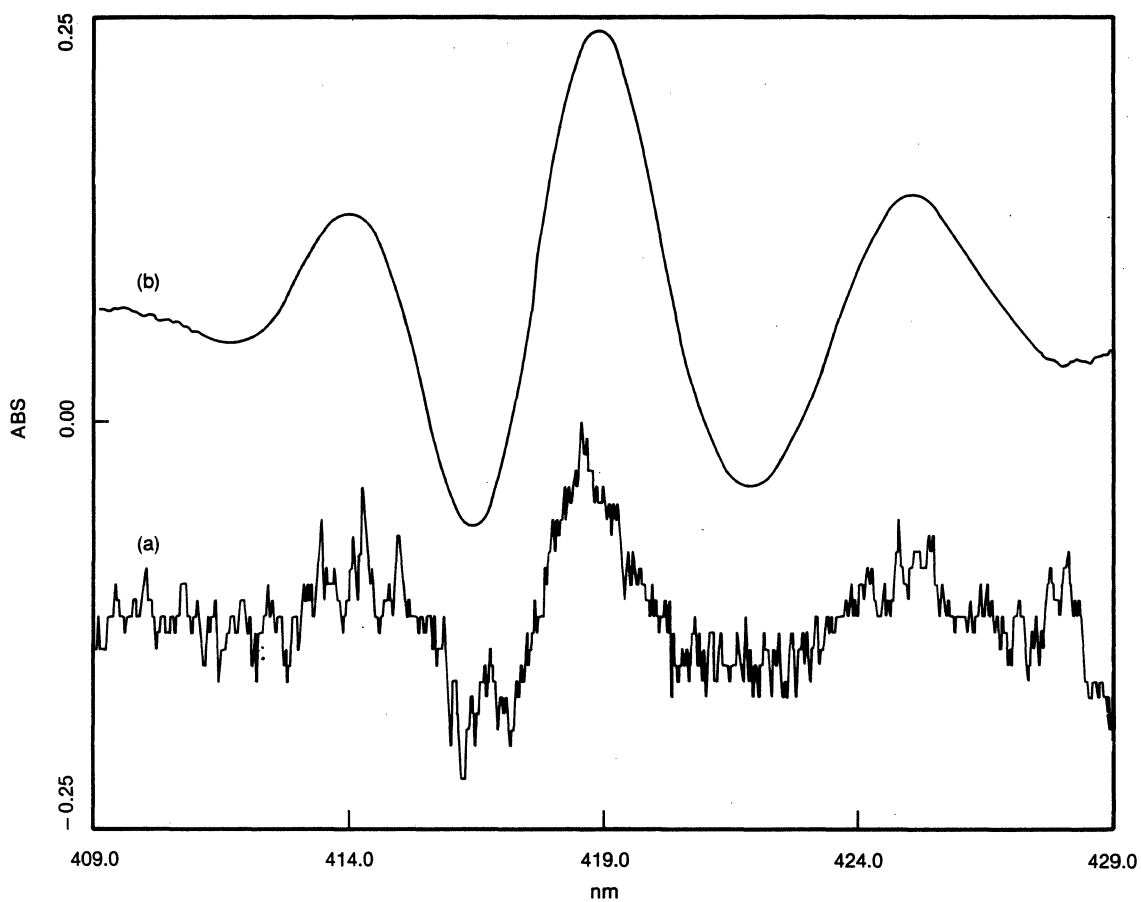


Figure 9-11. Fourth Derivative of Holmium Oxide Spectrum

-
4. To calculate the derivative of a spectrum stored in memory N1, and store the result in memory N2, enter the following command:

Memory N1 Diff K . M Transfer Memory N2 Enter

N1 and N2 are integers (0-4), specifying memory locations. K is an integer (1-4), specifying the order of the derivative. M is an integer (1-99), specifying the width of the sliding window. If an invalid number is entered for K or M the command will not execute properly. If this occurs, repeat the entire command using the correct values.

5. While the calculation is in progress, the red light on the measure key will be illuminated. When the calculation is complete, the light will go out.
6. Display the derivative spectrum on the CRT, using the following key sequence:

Memory N2 Enter

7. Derivative spectra are always displayed on a scale of ± 0.250 ABS, or $\pm 25.0\%$ T. The data can be scaled to fit within these limits by using the arithmetic operations to multiply or divide the data file by the appropriate constant.
8. If the noise level in the derivative spectrum is too high, increase the value used for M and repeat step 4.
9. If the spectral features of the derivative appear distorted or shifted along the wavelength axis, decrease the value used for M and repeat step 4.
10. A copy of the derivative spectrum can be plotted on the thermal printer/plotter, as with any other spectrum, using the following command:

Memory N2 Measure

See Section 9.4 for details on overlay plotting.

9.8 PEAK PICKING

9.8.1 DESCRIPTION

The peak-picking software automatically locates peaks in either absorbance or transmittance spectra. A listing of the peak position and the photometric value at the peak is provided on the printer/plotter.

A noise sensitivity value (NSV) can be specified to avoid the selection of spurious peaks. Any peak with a height less than the NSV is ignored. Up to twenty peaks can be identified in a given spectrum. If more than twenty peaks are found, only the first twenty, starting from the higher wavelength side of the spectrum, are printed.

Peak heights are always measured from the top of the peak to the baseline valley on the higher wavelength side of the peak. The NSV should be less than or equal to the height of the smallest peak in the spectrum. An example is shown in Figure 9-12.

The peak labelled #5 was selected as the smallest peak of interest. The peak height measured to the valley on the higher wavelength side of the peak is 0.1 ABS, as shown in the figure. A slightly smaller NSV was selected. The results obtained from the peak-picking software, using NSV=0.09 ABS, are shown in Table 9-3.

Table 9-3. Peak-Picking Software Output (For Spectrum in Figure 9-12)

Noise sensitivity value = 0.0900

NO.	PEAK WAVELENGTH	PEAK VALUE
1	536.4 nm	0.270 ABS
2	460.2 nm	0.757 ABS
3	453.6 nm	0.665 ABS
4	445.8 nm	1.692 ABS
5	418.6 nm	0.211 ABS
6	361.0 nm	0.714 ABS

Note that the peak value shown in the table is the actual photometric value measured at the peak, and not the peak height measured by the program.

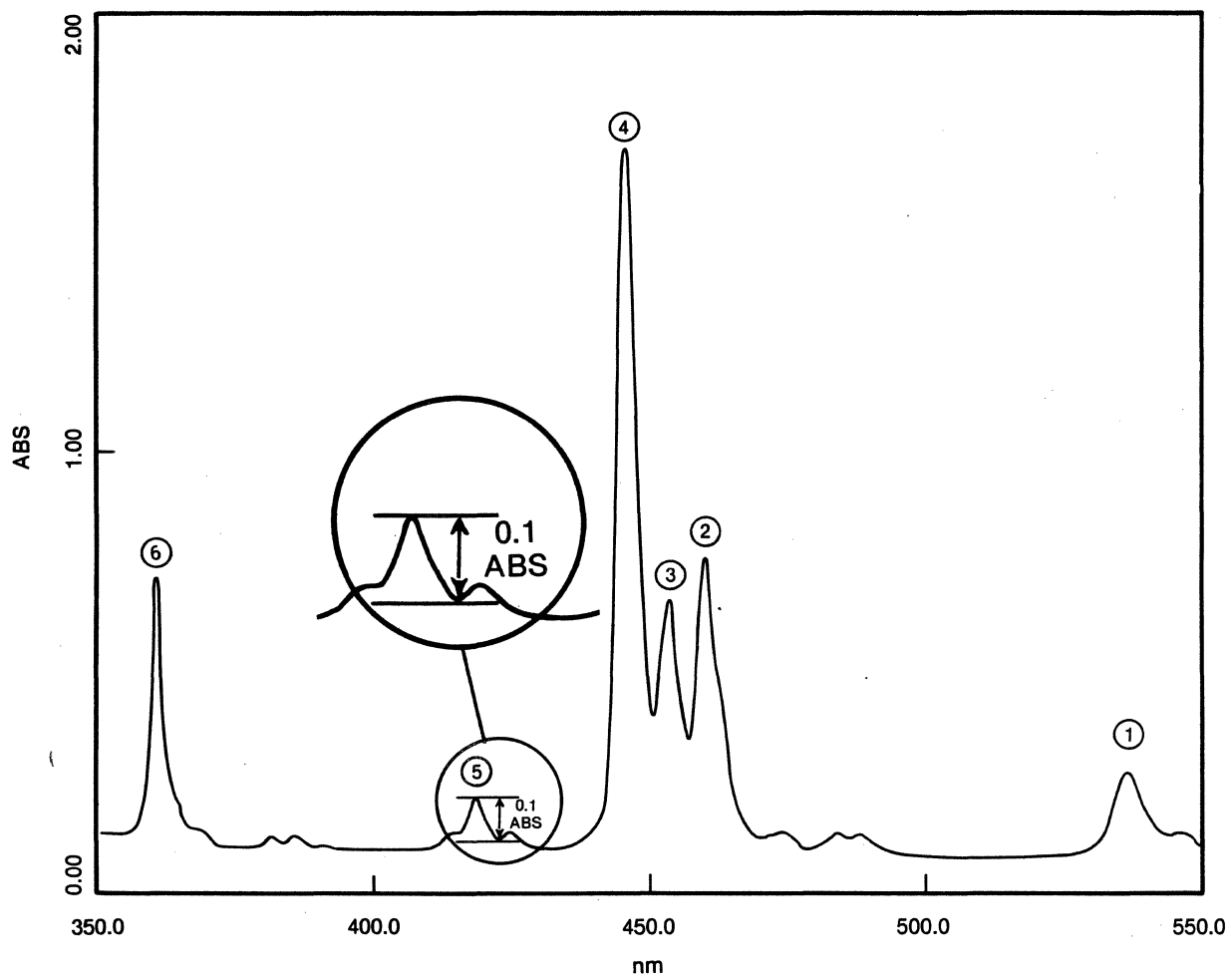


Figure 9-12. Calculation of Peak Height

Selecting smaller NSV's results in the selection of more peaks. Table 9-4 shows the results obtained when an NSV of 0.002 ABS was used on the same spectrum. Note that the number of peaks found increased from 6 to 15 as the NSV was decreased from 0.09 ABS to 0.002 ABS. Figure 9-13 shows an expanded scale plot of the same absorbance spectrum, indicating the position of the additional peaks.

Table 9-4. Effect of Smaller NSV on the Peak-Picker Output

Noise sensitivity value = 0.0020

NO.	PEAK WAVELENGTH	PEAK VALUE
1	546.4 nm	9.131 ABS
2	536.4 nm	0.270ABS
3	488.2 nm	0.131 ABS
4	484.2 nm	0.135 ABS
5	474.0 nm	0.138 ABS
6	460.2 nm	0.757 ABS
7	453.6 nm	0.665 ABS
8	445.8 nm	1.692 ABS
9	424.8 nm	0.136 ABS
10	418.6 nm	0.211 ABS
11	391.0 nm	0.109 ABS
12	385.8 nm	0.132 ABS
13	381.6 nm	0.131 ABS
14	361.0 nm	0.714 ABS
15	353.0 nm	0.136 ABS

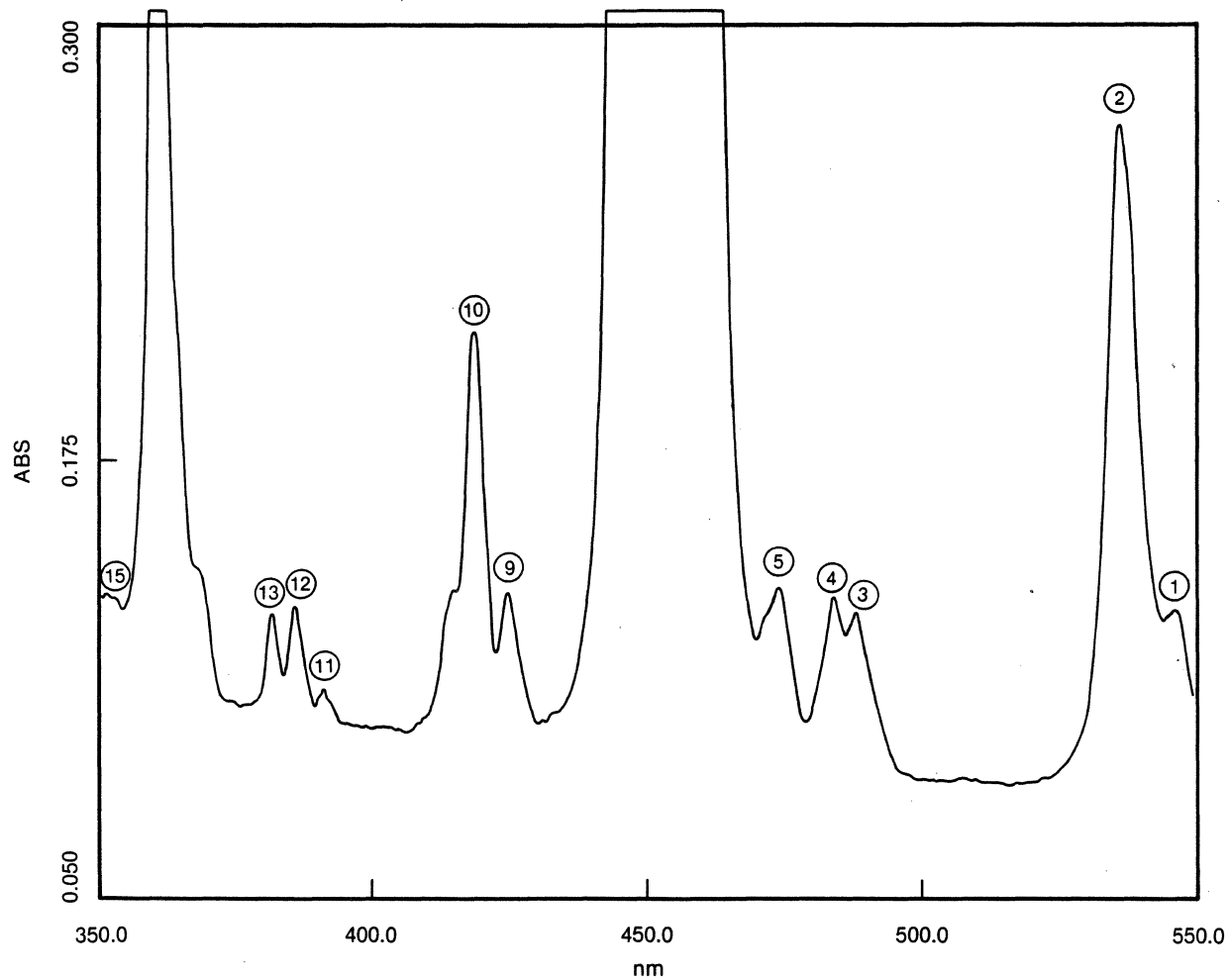


Figure 9-13. Expanded Scale Plot of Spectral Data used to Generate Table 9-4. Peaks 6, 7, 8 and 14 are offscale.

9.8.2 PROCEDURE

1. Display the spectrum to be analyzed on the CRT by pressing the following keys:

N1 is an integer (0-4) that indicates the memory location in which the data is stored. The peak picker can be used on derivative spectra as well as on absorbance or transmittance spectra.

2. Determine the minimum peak height that should be selected by the peak picker. The peak height is measured from the top of the peak to the baseline valley on the higher wavelength side of the peak (see Figure 9-12).

In an absorbance spectrum, the peak is measured at an absorbance maximum. In a transmittance spectrum, the peak is measured at a transmittance minimum.

3. Select a noise sensitivity value (NSV) that is slightly smaller than the minimum peak height desired, and enter the NSV using the following key sequence:

The units used for the NSV are either %T or ABS. The results are automatically printed out as shown in Tables 9-3 and 9-4.

4. If necessary, repeat step 3 with larger or smaller NSVs. Larger NSVs will result in the selection of fewer peaks. Smaller NSVs will result in the selection of more peaks. The first 20 peaks located in a given spectrum will be printed.

9.9 SUMMARY OF METHOD 11 COMMANDS

The following examples summarize the commands used in Method 11. For full details, see the appropriate sections of this manual.

1. Transfer and exchange of data files (Section 9.2) :

Memory N1 Transfer Memory N2 Enter ,
Memory N1 Exch Memory N2 Enter

2. Displaying stored spectra (Sections 9.3, 9.4) :

Memory N1 Enter (for CRT),
Memory N1 Measure (for printer/plotter).

3. Arithmetic operations (Section 9.5) :

a) with constants

Memory N1 x, ÷, +, - Numeric Value Transfer
Memory N2 Enter ,

b) between spectra

Memory N1 x, ÷, +, - Memory N2 Transfer
Memory N3 Enter

4. Smoothing and differentiation (Sections 9.6, 9.7) :

Memory N1 Smooth M Transfer Memory N2 Enter ,
Memory N1 Diff K . M Transfer Memory N2 Enter

5. Peak Picking (Section 9.8) :

$\Delta\lambda$

Noise Sensitivity Value

ENTER

10.0 METHOD 51 - LIGHT SOURCE OPERATION AND CHANGEOVER

10.1 DESCRIPTION

Two lamps are used as radiation sources in the 9420/30. The tungsten (W) and deuterium (D₂) lamps cover the wavelength region from 910-340 and from 340-185 nm respectively.

Under normal operating conditions, both lamps are on at all times. A mirror is used to direct the light from the appropriate lamp into the monochromator. The mirror flips automatically at 340 nm.

Using method 51, these normal operating conditions can be changed. Either or both of the lamps can be turned off. This feature is utilized if only one lamp is needed for the spectral range being used. By turning off the unused lamp, its lifetime can be extended. The ability to turn off the lamps is also useful in testing and diagnostics.

The wavelength at which lamp changeover occurs can be varied within the region 340 to 350 nm.

10.2 PROCEDURE

1. Press the **Method** , **5** , **1** and **Enter** keys. The CRT will appear as shown in Figure 10-1.

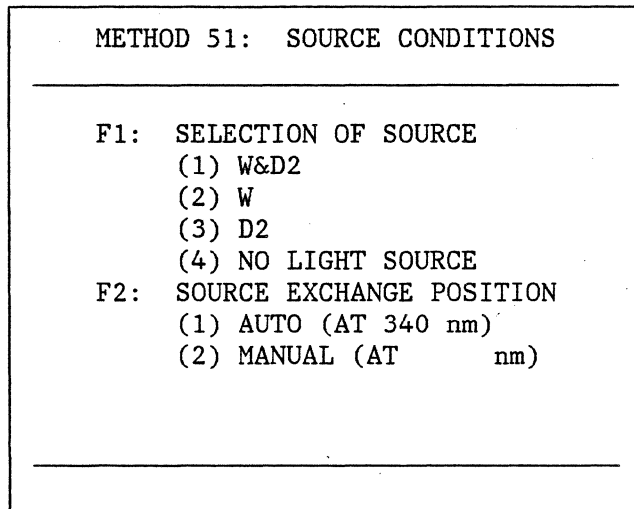


Figure 10-1. Method 51 Display

Two sets of conditions are presented for spectrophotometer operation. The first set (F1) selects the lamps to be used. The second set (F2) determines the wavelength at which the source exchange is to occur.

2. To change the selection of the source, press the number **1**. One of the following status indications will appear at the bottom of the CRT:

- (1): W and D2 (both lamps on).
- (2): W (tungsten lamp on).
- (3): D2 (deuterium lamp on).
- (4): NO LIGHT SOURCE (both lamps off).

To select a new condition, press the numeric key that corresponds to the desired condition, and **Enter**. The new condition will appear on the bottom of the screen, and this condition will remain in effect until another one is chosen. The preset condition may also be reinstated by use of Method 53 (see Section 12.0).

3. To change the source exchange position, press the number **2**. One of the following status indications will appear at the bottom of the CRT:

- (1): AUTO (at 340 nm).
- (2): MANUAL (AT---.---nm).

To select a new condition, press the corresponding numeric key, and Enter. Condition (1) is the preset condition that programs the instrument for a single changeover wavelength of 340 nm. If condition (2) is selected, the CRT will display a prompt for selection of the new changeover wavelength as shown below:

0.0

```
(2)MANUAL( nm)340.0 ~ 350.0 nm |
```

Using the numeric keys, enter the new wavelength (e.g., 343.0 nm). As the numbers are entered, they will be displayed in the window of the CRT. Press Enter to complete the change.

11.0 METHOD 52 - BEEP, PRINTER/PLOTTER, PEN POSITION AND DECIMAL POINT CONTROL

11.1 DESCRIPTION

Method 52 allows the following operations to be performed:

1. Control of the audible "beep" of the keypad.
2. Adjustment of the thermal printer head position.
3. Selection of the mark search mode on the printer/plotter.
4. Expansion of the photometric display from four digits to five digits.

The procedure used for each operation is discussed separately in the following sections.

11.2 PROCEDURE

1. Press the **Method** , **5** , **2** and **Enter** keys. The CRT will display the selections as shown in Figure 11-1.

METHOD 52: CONTROL SETTINGS

F1: BEEP
(1): ON
(2): ON (FOR KEY INPUT ONLY)
(3): OFF

F2: THERMAL HEAD ADJUST
(1): NORMAL
(2): FULL SCALE POSITION (WITH PLOT)
(3): 0 POSITION (WITH PLOT)
(4): FULL SCALE POSITION (WITHOUT PLOT)
(5): 0 POSITION (WITHOUT PLOT)

F3: MARK SEARCH
(1): ON
(2): OFF

F4: DIGITAL DISPLAY
(1): 4-DIGITS
(2): 5-DIGITS

Figure 11-1. Method 52 Display

2. To change the response of the audible "beep", press the number . One of the following status indications will appear at the bottom of the screen:

(1): ON
(2): ON (FOR KEY INPUT ONLY)
(3): OFF

To select a new condition, press the appropriate numeric key, and .

The normal state is (1) ON. In this mode, the instrument will "beep" once each time a valid key is pressed. The instrument will "beep" three times if an invalid key is pressed, or if an error has occurred (see Section 2.3.2).

If the (2): ON (FOR KEY INPUT ONLY) mode is selected, the three "beep" error message is disabled.

If the (3): OFF mode is selected, the "beep" is disabled completely.

3. To adjust the thermal head position at zero or full scale, press the number **2** . The message (1): NORMAL will appear at the bottom of the CRT. The options available are described below.

- a. (2): FULL SCALE POSITION (WITH PLOT) - If the number **2** is pressed, the thermal head will move to the full scale position, and the chart paper will begin to advance slowly. A line is automatically drawn at the position of the thermal head, in a strip-chart recorder like mode. To adjust the head position, use the SPAN adjustment on the printer/plotter (see Figure 1-4). A small screwdriver is supplied in the instrument tool kit for making this adjustment.

When the adjustment is complete, press **Stop** . The system will automatically return to the (1): NORMAL state.

- b. (3): 0 POSITION (WITH PLOT) - If the number **3** is pressed, the thermal head will deflect to the zero position, the chart will begin to advance slowly, and a line will be drawn automatically at the head position. To adjust the head position, use the ZERO adjustment on the printer/plotter (see Figure 11-1).

When the adjustment is complete, press **Stop** . The system will automatically return to the (1): NORMAL state.

- c. (4): FULL SCALE POSITION (WITHOUT PLOT) - This condition is identical to number (2), except that the chart paper will not advance, and no line is drawn. Again the Stop command is used when the adjustment is complete.
- d. (5): 0 POSITION (WITHOUT PLOT) - This condition is identical to number (3), except that the chart paper will not advance, and no line is drawn. As in the other steps, the Stop command is used when the adjustment is complete.

4. The MARK SEARCH mode is accessed by pressing the number **3** . One of the following status indications will appear on the bottom of the CRT:

- (1): ON.
(2): OFF.

To change the status, press the appropriate numeric key, and **Enter** .

If (1): ON is selected, the printer/plotter will search for the first line on the gridded chart paper before beginning to write. The normal condition is (1): ON.

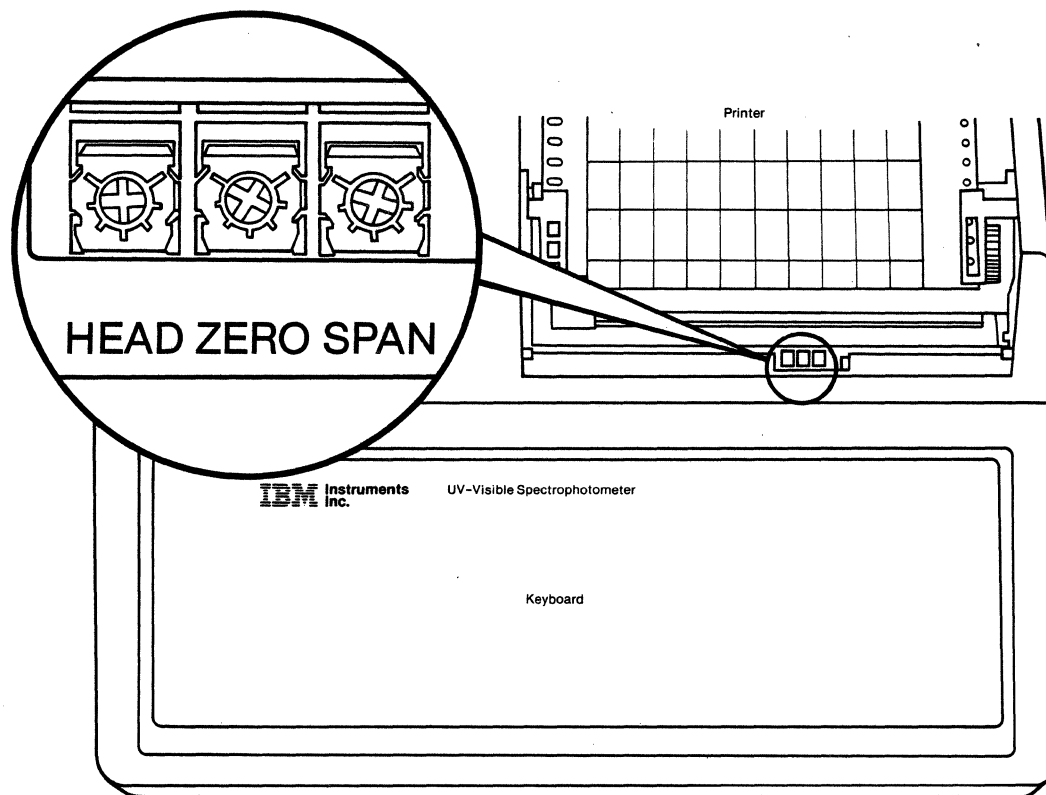


Figure 11-2. Thermal Printer Controls.

5. To change the number of digits displayed on the CRT for the measured photometric values, press the number 4 . One of the following status indications will appear on the bottom of the CRT:

- (1): 4-DIGIT.
- (2): 5-DIGIT.

To select a new condition, press the appropriate numeric key and Enter .

The normal condition is (1): 4-DIGIT. In the four-digit mode, one decimal place is shown in %T readings and three decimal places are shown in ABS readings (e.g., 100.0 %T and 1.000 ABS). In the five digit mode, an extra decimal place is displayed for both photometric readings (e.g., 100.00 %T and 1.0000 ABS).

12.0 METHOD 53 - INITIALIZATION

12.1 DESCRIPTION

Three functions are provided in Method 53:

1. Reinitialization of all parameters.
2. Recorrection of the baseline.
3. Input of the date.

12.2 PROCEDURE

1. Press the **Method**, **5**, **3** and **Enter** keys. The CRT will appear as shown in Figure 12-1.

```
METHOD 53:  INITIALIZATION
-----
F1:  PARAMETER INITIALIZE
     (1):  ALL CLEAR
F2:  INITIAL BASE LINE CORRECTION
     (1):  ON
     (2):  RECORRECTION
     (3):  OFF
F3:  DATE INPUT
     (1):  OFF
     (2):  ON
     (3):  INPUT (MM DD YY)
-----
```

Figure 12-1. Method 53 Display

2. To reinitialize the parameters in all the methods, press number **1**. The following prompt will appear on the bottom of the CRT:

(1): ALL CLEAR.

At this point, the procedure can be aborted by pressing **Stop**. This prevents accidental clearing. To carry out the initialization, press **1** and **Enter**. All instrumental conditions will be returned to their original state (the state they assume when the instrument is first powered up).

3. To recorrect the baseline, press **2**. One of the following status indications will appear on the bottom of the CRT:

(1): ON.
(3): OFF.

If a baseline was stored during the power-up initialization procedure (see Section 2.2.2), it will be used during all spectral measurements, as long as the (1): ON state is selected. If the (3): OFF state is selected, the stored baseline information is not used.

If a new baseline is desired, press **2**. The message RECORRECTION UNDER WAY will appear at the bottom of the CRT. Recorrection takes approximately ten minutes. When the recorrection is complete, the instrument will automatically enter the (1): ON state.

Note: If a recorrection is done, be sure that both lamps are turned on (see Method 51, Section 10.0).

4. To enter the date mode, press the number **3**. One of the following status indications will appear at the bottom of the CRT:

(1): OFF.
(2): ON.

To enter the date, press **3**. A prompt will appear at the bottom of the CRT:

(3): INPUT (MM DD YY).

The date is entered as six numbers, the first two for the month, the next two for the day and the last two for the year. For example, Sept. 30, 1983 would be entered as 093083. Press **Enter** to complete the entry. The system will automatically enter the (2): ON state. The date will be displayed at the upper left corner of the CRT, and will appear in any headers created using the **Print List** key.

13.0 METHOD 54 - PHOTOMULTIPLIER TUBE VOLTAGE CONTROL

13.1 DESCRIPTION

Under normal operating conditions (double-beam mode), the 9420/30 automatically controls the photomultiplier tube (PMT) voltage. The voltage is varied as a function of wavelength to provide excellent compensation for changes in source intensity, without affecting the resolution of the scan.

The 9420/30 can also be used in a single-beam mode. This mode is often used to look at the profile of the source lamps, and to test the resolution and wavelength accuracy of the instrument.

In the single-beam mode, the automatic control of the PMT voltage cannot be done, so a fixed PMT voltage must be selected. Method 54 provides the ability to enter the single-beam mode, and to fix the photomultiplier voltage. The new voltage is applied after the program exits from Method 54.

13.2 PROCEDURE

1. Press **Method** , **5** , **4** and **Enter** keys. The CRT will appear as shown in Figure 13-1.

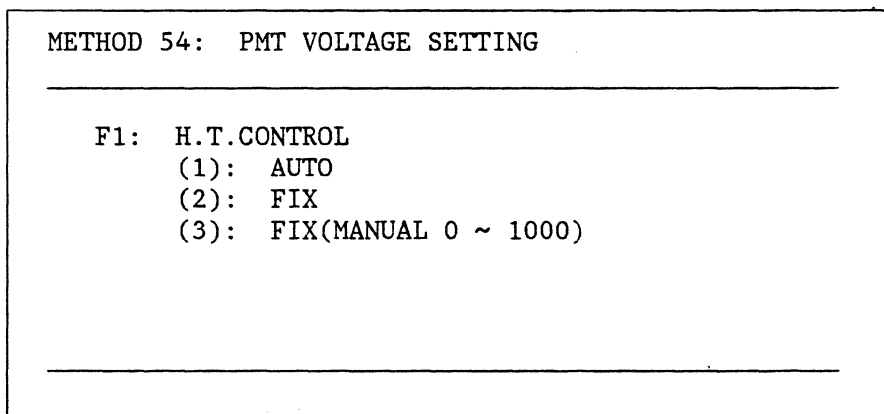


Figure 13-1. Method 54 Display

2. To change the PMT voltage setting, press **1** . One of the following status indications will appear at the bottom of the CRT:

- (1): AUTO.
- (2): FIX.
- (3): FIX(MANUAL 0 ~ 1000).

To select a mode, press the correct numeric key, 1, 2 or 3 and **Enter**. The three settings are described below:

- a. (1): AUTO - This is the normal operating mode. Selecting AUTO automatically puts the instrument in the double-beam mode, and controls the PMT voltage so as to normalize the response of the reference channel.
 - b. (2): FIX - This mode places the instrument in the single-beam mode. The PMT voltage is automatically set at 300 V.
 - c. (3): FIX(MANUAL 0 ~ 1000) - This mode also places the instrument in the single-beam mode. When **3** is pressed, a prompt will appear at the bottom of the CRT, indicating that a voltage should be entered. Using the numeric key pad, select a voltage between 0 and 1000. Initially, the voltage is set at 300V. Any change will not take effect until the Method key is used to leave Method 54.
3. The single beam mode is used by the instrument to check the wavelength accuracy and bandwidth specifications. The following procedure is used.
- a. Using Method 54, select a fixed PMT voltage of 525 volts, as described in step 2 above.
 - b. Using Method 51, select only the deuterium source (F1, option 3, see section 10.2 for details).
 - c. Using Method 1, scan the region from 658.6 nm to 653.6 nm. The following conditions are recommended:

- λ Scale = 0.5 nm/cm
- Scan speed = 4 nm/min
- %T/Abs = %T
- %T Abs Scale = 0-100% T
- Bandwidth = 0.1 nm

A sharp peak at λ max = 656.1 nm should be obtained, as shown in Figure 13-2.

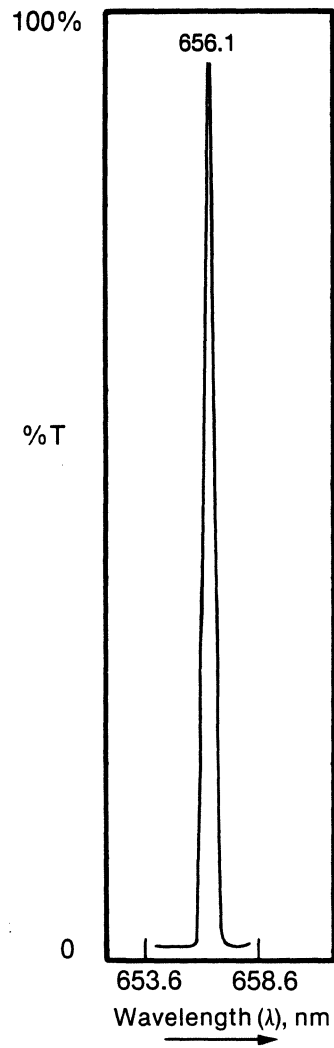


Figure 13-2. Deuterium Emission Line (λ max = 656.1 nm).



14.0 METHOD 55 - SPECTRAL SUBTRACT/RATIO MODE

14.1 DESCRIPTION

The 9420/30 can be used to automatically calculate ratio spectra in transmittance mode and difference spectra in absorbance mode. This feature provides a versatile means for performing background correction under a variety of experimental conditions. This can be particularly useful when special accessories are used, or when the instrumental parameters are greatly different from those used during the standard baseline correction routine (described in section 2.2.2).

Method 55 is used to collect and store the background spectrum, S2, and to turn on the ratio/subtraction feature.

Method 1 is then used to collect the ratio or difference spectrum. As the sample spectrum, S1, is collected, the ratio or difference is automatically calculated, as shown below.

$$\begin{array}{l} \text{Difference Spectrum (ABS mode)} = S1 - S2 \\ \text{Ratio Spectrum (\%T mode)} = S1 \div S2 \end{array}$$

The ratio or difference spectrum is displayed on the CRT as the data is collected, and is stored in Memory 1 like any other Method 1 spectrum.

Note: The uncorrected spectrum, S1, is not displayed or stored in memory, as long as the Method 55 feature is on.

14.2 PROCEDURE

1. Set the data collection parameters for the experiment using Method 1.
2. Press the **Method** , **5** , **5** and **Enter** keys. The CRT will display the Method 55 menu as shown in Figure 14-1

METHOD 55: SPECTRAL SUBTRACT/RATIO MODE

F1: DIFFERENCE OR RATIO SPECTRUM (ONLINE)
(1) OFF
(2) ON
(3) COLLECTION

Figure 14-1. Method 55 Display

3. Place the sample which will provide the background spectrum, S2, into the sample chamber.
4. Press the number key. The current status of Method 55, ON or OFF, will be displayed at the bottom of the CRT.
5. Press the and keys. A message will appear at the bottom of the CRT indicating that DATA COLLECTION is underway. The background spectrum is collected and stored in Memory 0. When data collection is complete, the Method 55 status is automatically set to ON.

Note: Since Method 55 uses Memory 0 for data storage, Memory 0 should not be used for routine data handling when Method 55 is ON.

6. To collect the ratio or difference spectrum, return to Method 1 by pressing the , and keys. The message #55 ON will appear at the top left of the CRT as a reminder that the ratio/subtraction feature is activated.
7. Place the sample in the sample chamber, and press . The ratio or difference spectrum is displayed on the CRT and stored in Memory 1.
8. To turn off the ratio/subtraction feature, return to Method 55 and press the , and keys. The instrument will return to normal operation.

A.0 9420/9430 ERROR CODES

A.1 DIAGNOSTIC ERRORS

If one of these errors occurs during initialization, refer to the troubleshooting guide in Appendix C.

- E001: Wavelength comes too far to short side.
- E002: Wavelength comes too far to long side.
- E003: Wavelength drive mechanism is not functioning.
- E004: Sector motor is not functioning.
- E005: Slit drive motor is not functioning.
- E006: Tungsten lamp is off.
- E007: Deuterium lamp is off.
- E008: Slit width is too large.
- E013: Malfunction of source exchanging mirror (Tungsten lamp side).
- E014: Malfunction of source exchanging mirror (Deuterium lamp side).
- E015: Defective RAM.
- E019: %T/ABS conversion is not proper.

A.2 PARAMETER ERRORS

If one of these errors occurs during operation, check the indicated parameters and adjust them accordingly. See Appendix C for more details.

- E010: The data specified for data processing do not have the same photometric mode, %T or ABS.

-
- E011: The data specified for data processing do not have the same " λ scale" value.
 - E012: The data specified for data processing do not have the same " λ SET" value.
 - E020: Improper combination of λ scale and λ set.
 - E021: Improper combination of λ scale and scan speed.
 - E101: The data collection parameters (%T/ABS mode, λ Scale and λ Set) selected for Method 1 do not match the parameters used to collect the reference spectrum store in Memory 0. Method 55 is in the "on" state.

B.0 LAMP REPLACEMENT PROCEDURE

B.1 SAFETY CONSIDERATIONS

The following safety precautions should be taken when replacing lamps:

1. Electrical Safety

Hazardous voltages are used in the lamp section. Therefore the instrument should be turned off and unplugged during the replacement part of this procedure.

2. Exposure to Radiation

When the deuterium (D_2) lamp is on, it emits ultraviolet radiation which is harmful to the eyes. Therefore, eye protection must be worn. The use of goggles which strongly attenuate the ultraviolet is recommended.

3. Lamp Handling

Do not handle the lamps directly with bare hands. A lamp contaminated with body oils should be rinsed in a solvent (hexane) and wiped dry (not air dried).

Note: Do not touch any optical component, i.e mirrors, gratings, etc. This will invalidate the warranty.

B.2 LAMP ACCESS AND IDENTIFICATION

1. Removal of the Lamp Housing Cover

The lamp housing, located at the right rear corner of the Optical Unit is shown in Figure B-1 (top view from front).

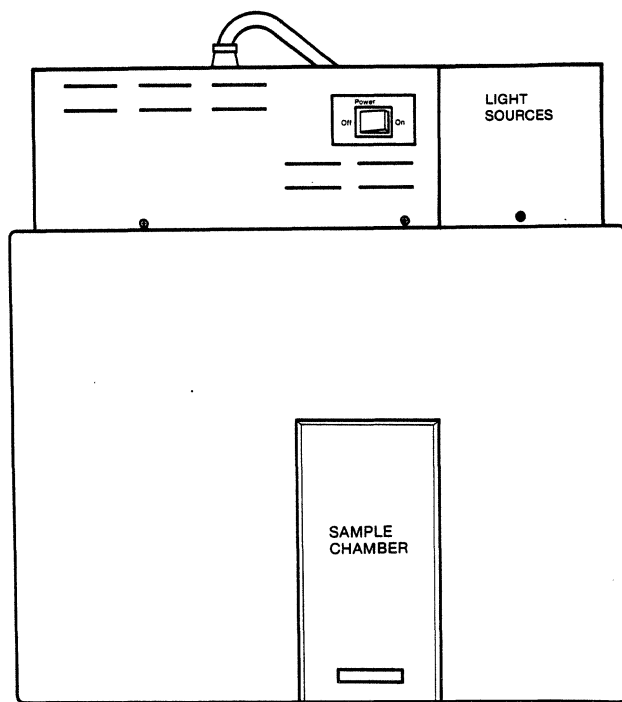


Figure B-1. Location of Lamp Housing

To remove the cover, loosen the thumb screw and back the screw out several turns. Gently rock and raise the rear of the cover enough to clear the shoulder screws. Slide it rear-ward, and lift it off.

2. Identification of the Lamps

The location of the individual lamps within the housing is shown in Figure B-2 (top view from rear).

The W Lamp is located close to the left side of the instrument, as indicated by "W" in Figure B-2. It is identified by its spherical bulb, small tungsten filament, a mirror coating on a large spot on the glass behind the filament, and two electrical connections. The D₂ lamp is located to the right of the W lamp. It is identified by its cylindrical bulb clamped into a special mounting assembly, a small rectangular aperture in the metal assembly inside the bulb, and three electrical connections. The third component is the lamp selecting mirror.

Note: Do not touch the mirror. Fingerprints on the mirror will greatly degrade the instrument performance.

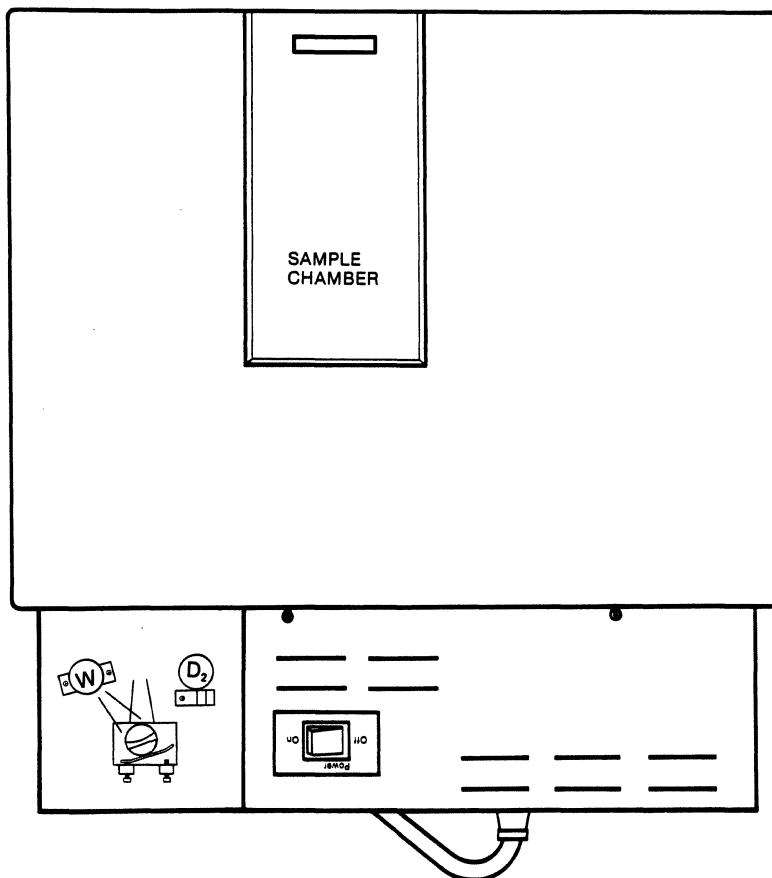


Figure B-2. Location of Lamps

B.3 LAMP REPLACEMENT

1. The W Lamp

Disconnect the electrical connector to the W lamp. Unlock the lamp by rotating it counter clockwise (CCW), and lift it out. Do not touch the lamps with bare hands.

Place the new W lamp on the holder with the mirror behind the filament, the leads down through the bottom slot of the lamp mount and out toward the mating connector. Seat it on the three locking pins and rotate it CW until it is fully locked. Connect the electrical connector.

2. The D₂ Lamp

Disconnect the electrical connector to the D₂ lamp. Remove the entire lamp mounting assembly by removing the one screw which holds it to the casting (see Figure B-3) and lifting the assembly from the two locating pins.

After noting the position of the aperture in the clamp, loosen the clamp which holds the lamp in the assembly. Slide the old lamp out and the new one in. Position it approximately the same as the old one was (with the internal aperture aimed toward mirror M₁). Leave the clamp loose enough for adjustment. Replace the assembly on the locating pins, and retighten the screw to the casing. Connect the electrical connector.

B.4 OPTICAL ALIGNMENT

1. General

Caution: In this section the lamps will be powered on with the cover off. Read carefully the safety considerations in section B-1 before proceeding.

Turn the instrument on. Using the Source Control Method 51 each lamp is turned on individually (one at a time) to eliminate interference with visual inspection. Refer to Figure B-3 for an alignment diagram of the light source optics, viewed from behind the instrument.

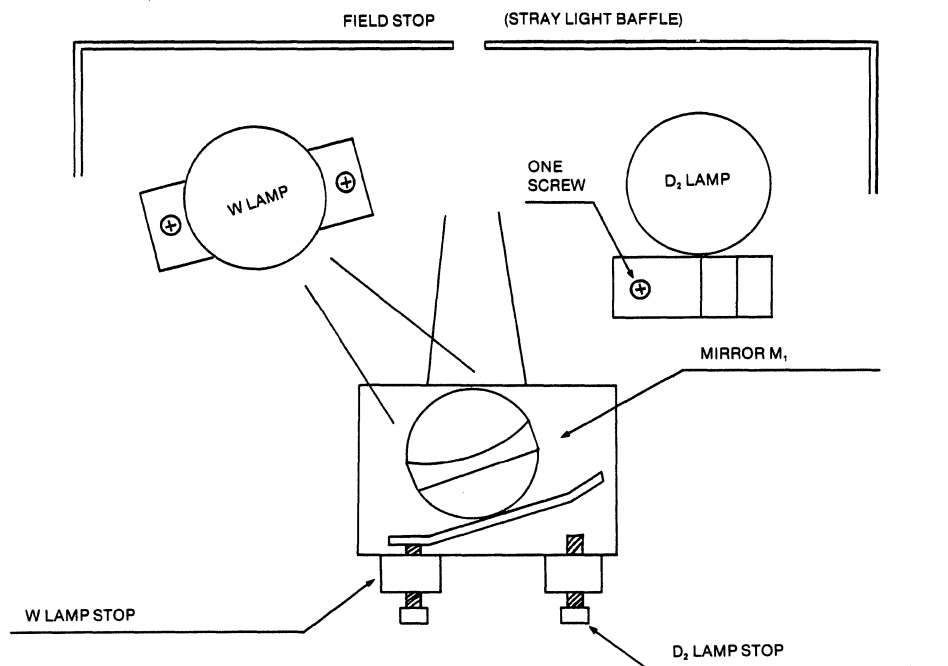


Figure B-3. Alignment Diagram

The spherical mirror, M_1 , images one or the other of the lamps onto the field stop (a rectangular opening in the wall leading to the monochromator). The "lamp stop" is a mechanical stop used to center the image horizontally at the field stop. This is done by first loosening the lower socket head screw of the clamping mechanism for the adjustment (use a 2.5mm hex key from tool kit provided). The upper screw is then adjusted to center the image and the lower screw is then retightened to clamp the adjustment. (The clamping mechanism affects the adjustment slightly and should be taken into account in the adjustment; the alignment should be rechecked after clamping).

2. Alignment of the W Lamp Optical Path

Use Source Control Method 51 to turn on the W lamp alone. The horizontal alignment is accomplished as in Step 1 above.

The vertical dimension should not need adjustment.

3. Alignment of the D₂ Lamp Optical Path

Use Method 51 to turn on the D₂ lamp alone. Align the lamp itself as the first step in this procedure. It must be aligned in both the horizontal (angular) and vertical (axial) dimensions, based on the image position at the field stop, as in paragraph 4.1. The vertical position should be as accurate as possible. After alignment of the D₂ lamp, carefully tighten the clamp. The D₂ lamp stop (See Figure B-3) can be used for a fine adjustment of the horizontal alignment, if necessary, as with the W lamp. Use the Source Control Method 51 to return the instrument to operation with both lamps.

4. Finer Adjustments

Finer adjustments are made by using the PMT detector in the single beam mode, to maximize the light level. A PMT Voltage command (Method 54) is used to set the PMT voltage to a level determined experimentally to give a %T reading of about 80%. Initially the PMT voltage will be set to 300V. Adjust this voltage up or down to obtain a reading of about 80%T. Do not hold the voltage at a level which gives a reading greater than 150%T, or PMT damage could result. The voltage change does not become effective until Method 54 is exited, so it is best to move to Method 1 to check the %T reading after each change in the voltage. Selection of one lamp or the other is made simply by selecting a wavelength above or below 340nm. This alignment is made based on maximizing the %T reading. Bandwidth should be set between 2 and 5nm. Upon completion of this alignment procedure, return the voltage level on the PMT to its usual automatic setting.

C.0 TROUBLESHOOTING GUIDE

The following troubleshooting guide is intended to help correct simple malfunctions which might occur in the operation of the 9420 or 9430 Spectrophotometer.

If further assistance is needed, call your IBM Service Representative at one of the following toll-free numbers:

(800) 243-9435
(800) 952 -1073 (Connecticut Only)

1. Symptom: The instrument does not properly begin the initialization and diagnostic procedure when the power is turned on. Proper initialization is described in Section 2.2.

Corrective Action:

- a. Check all cable connections as described in Section 1.5. Ensure that the optical unit is plugged into a working electrical outlet.
 - b. Unplug the instrument from the electrical outlet, and check the power fuse, in both the optical unit and the operator's console. If a fuse has blown, replace it with another fuse of identical rating (current and voltage). If the fuse blows repeatedly when the power is turned on, call your service representative.
2. Symptom: The CRT fails to display the power-up message as shown in Figure 2-1.

Corrective Action:

- a. The power cord of the operator's console should be plugged into the power outlet on the black panel of the optical unit.
Caution: Ensure that the power cord of the operator's console is not plugged directly into a wall outlet.
- b. Ensure that the power switch of the operator's console is always in the ON position.

-
- c. Ensure that the CRT cable is securely attached to the jack marked CRT on the rear panel of the operator's console.
 - d. Check the brightness adjustment control on the back of the CRT. If necessary, the brightness can be increased. Use the small screw driver supplied with the instrument tool kit to turn the control clockwise. Caution: Avoid excessive brightness. If this fails to resolve the problem, do not leave the control turned up to its maximum setting.

3. Symptom: During the power-up diagnostics, the error message E006 or E007 appears on the CRT.

Corrective Action:

- a. E006 and E007 indicate a failure of the tungsten and deuterium source lamp, respectively. Caution: When opening the lamp compartment to check the lamps, eye protection must be worn to prevent exposure to harmful UV radiation. Never touch the lamps with bare hands. Complete lamp replacement procedures are given in Appendix B.
- b. If both lamps are functioning, check the cell compartment to see if there is any obstruction of the light path. If a special cell is in place, ensure that the accessory has been properly installed, and that the cell is filled with an appropriate solvent. After checking the light path, repeat the power-up procedure.

4. Symptom: During the power-up diagnostics, one of the diagnostic errors listed in Appendix A.1 appears on the CRT.

Corrective Action:

- a. Check all instrument cabling as described in Section 1.5.
- b. Check the light path as described in paragraph 3b above.
- c. Repeat the power-up procedure. If the error recurs, call your service representative.

5. Symptom: During operation, the message E010, E011 or E012 appears on the CRT.

Corrective Action:

- a. These messages indicate an incorrect choice of scan parameters. A description of each error, with appropriate corrective action is given in Section 3.1, paragraph 3.

6. Symptom: During operation, the message E101 appears on the CRT.

Corrective Action:

- a. The data collection parameters, (%T/ABS Mode, λ Scale and λ set), selected for Method 1 do not match the parameters used to collect the reference spectrum in Method 55. Change the parameters used in Method 1 to match the data stored in Memory 0, or turn off the Method 55 option as described in Section 14.

7. Symptom: An extra decimal point appears in the photometric data display. This indicates insufficient light reaching the detector. The following corrective actions can be taken.

Corrective Action:

- a. Ensure that the appropriate light source is selected. Method 51 can be used to select the source.
- b. Ensure that the light beam is properly centered on both the sample and reference cells. To observe the light beam, set the wavelength to 510nm, with a bandwidth of 2nm. A small green rectangle should be observed when a piece of white paper is held in front of the cell window. It may be necessary to dim the room lights to see the beam.
- c. Increase the spectral bandwidth. If this corrects the problem, it may be necessary to work with the higher bandwidth for that particular application.
- d. Check the absorbance of the solvent in the region being scanned. If the absorbance is high, it may be necessary to change to a more appropriate solvent.

8. Symptom: The instrument keypad fails to respond.

Corrective Action:

- a. Ensure that the key being pressed is used in the current Method.
- b. Check to see if the instrument is performing another task. If the Measure key lamp is illuminated, a measurement is in progress and the system will not respond to another command, with the exception of STOP.
- c. If the keypad fails to respond at all, power down the instrument and restart it. If the problem persists, call your service representative.

-
9. Symptom: During operation, the message E201 appears on the CRT. NOTE: This error will only occur if the Parameter Storage Board is installed.

Cause:

- a. An attempt is being made to store parameters in a File which is already full. (In use).
- b. An attempt is being made to delete an empty File.

NOTE: These operations are related to the Parameter Storage Board Option.

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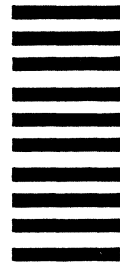
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